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The Journal of the Society of Dyers and Colourists

Volume 77 Number 10

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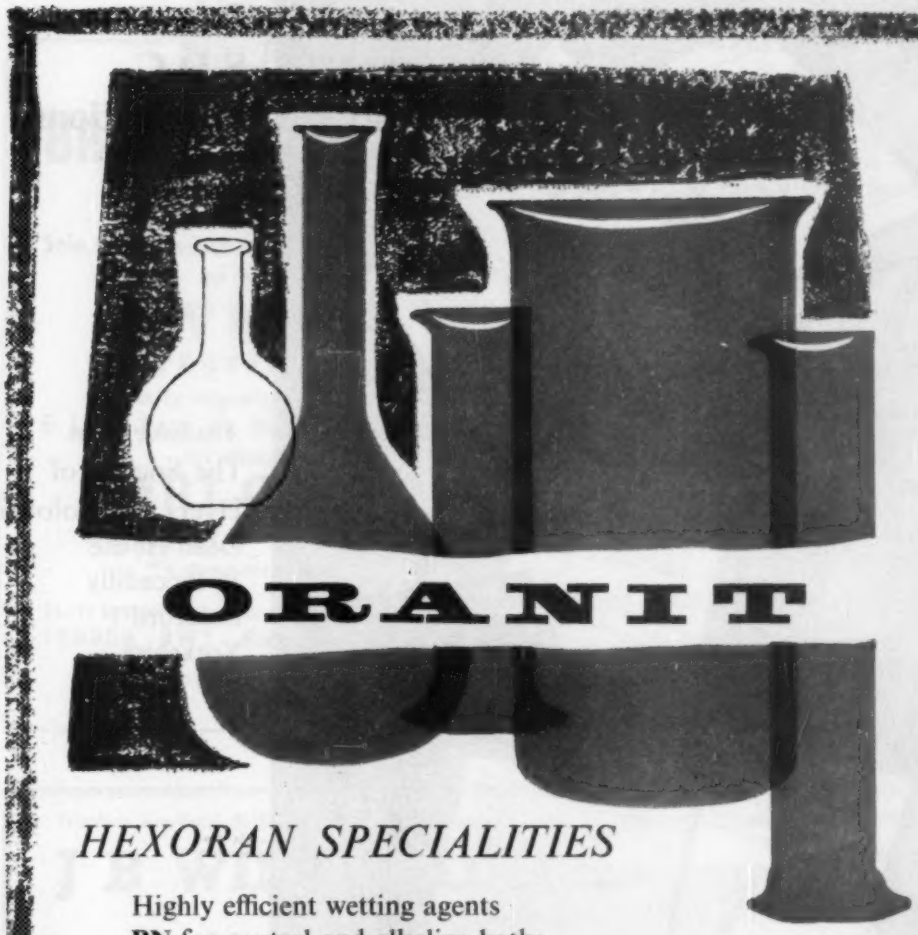

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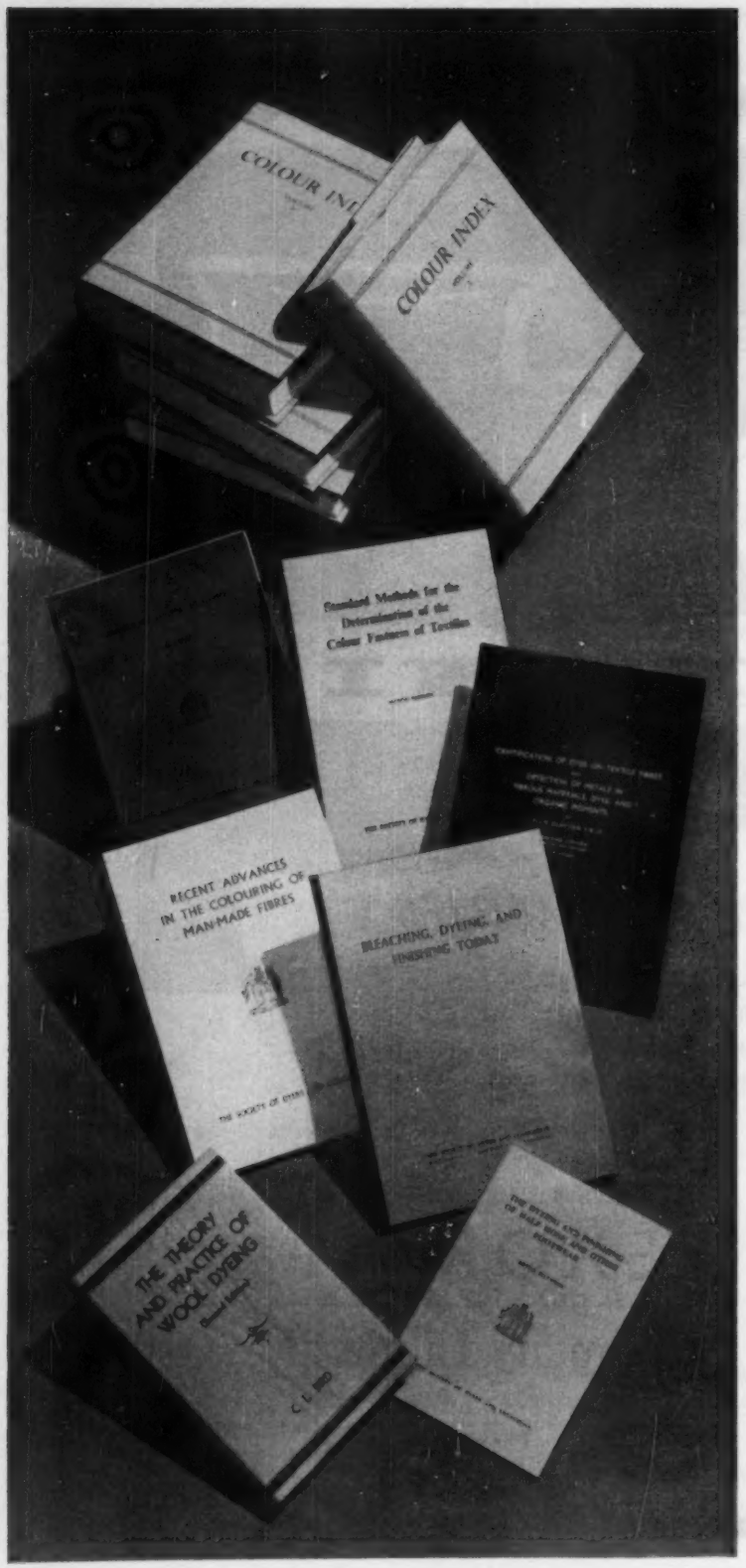
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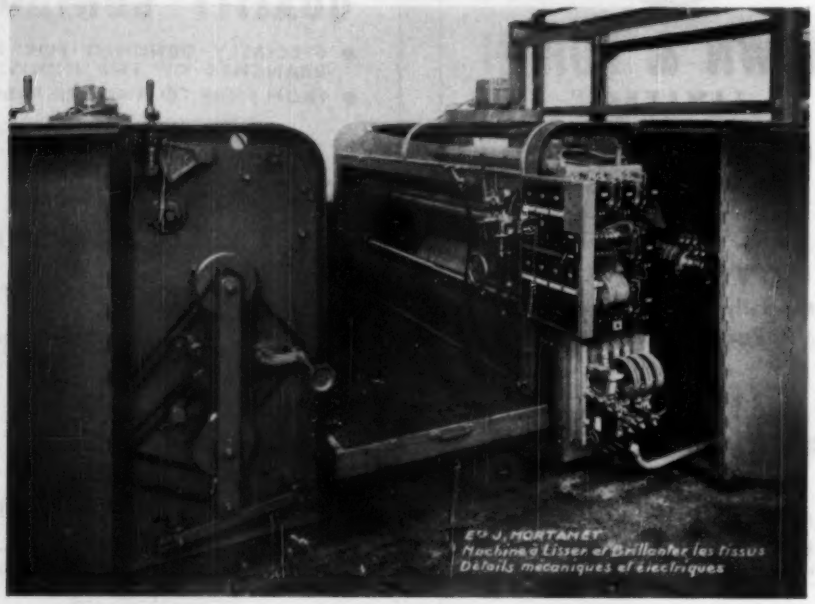

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Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Scouring and Dyeing Blends for the Carpet Trade	*R. C. Cheetham*
Dyeing and Finishing Terylene–Linen Fabrics	*I. E. Haden*
Legal and Practical Problems of Effluent Disposal	*J. H. Harwood*
Combined Dyeing and Finishing of Cellulosic Piece Goods	*I. D. Rattee*
An Introduction to Organic Pigments	*F. M. Smith*

COMMUNICATIONS

Cross-linking of Cellulose and its Derivatives I—Use of Unsaturated Esters in Cross-linking Reactions	*W. M. Corbett and J. E. McKay*
VI—Addition of Amines to Cellulose Crotonates	*J. E. McKay and W. Taylor*
Further Tests for Colour Vision	*F. Jordinson*
Photochemistry of Sodium Chlorite in Solution, with special reference to Chlorite Bleaching	*C. Kujirai and I. Fujita*
Sorption of Acids by Wool from Mixtures of the Acids I—Sorption of Hydrochloric Acid and Dichloroacetic Acid or Monochloroacetic Acid	*P. Larose and R. Donovan*
Paper Chromatography of Insoluble Dyes	*J. Sramek*
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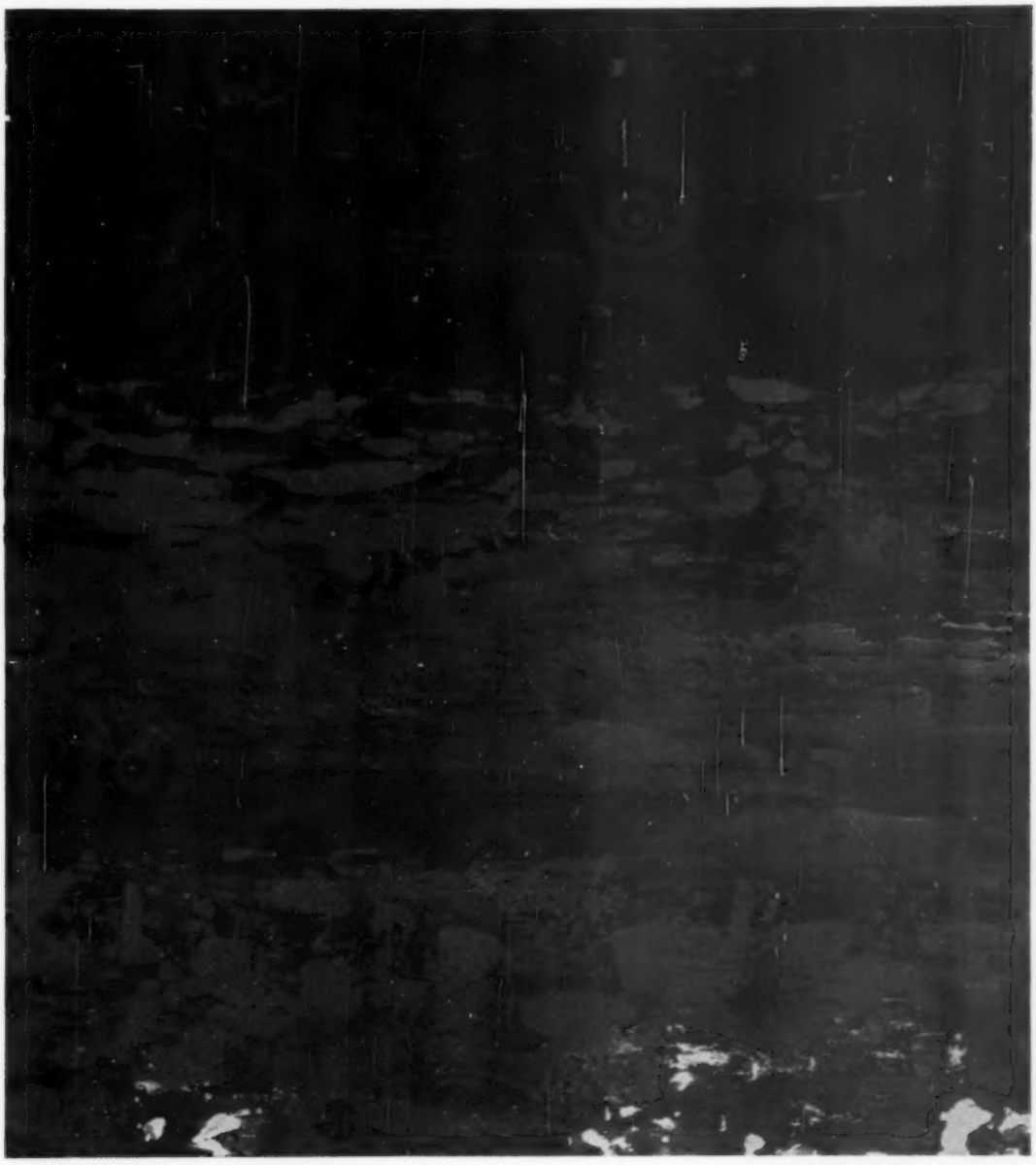

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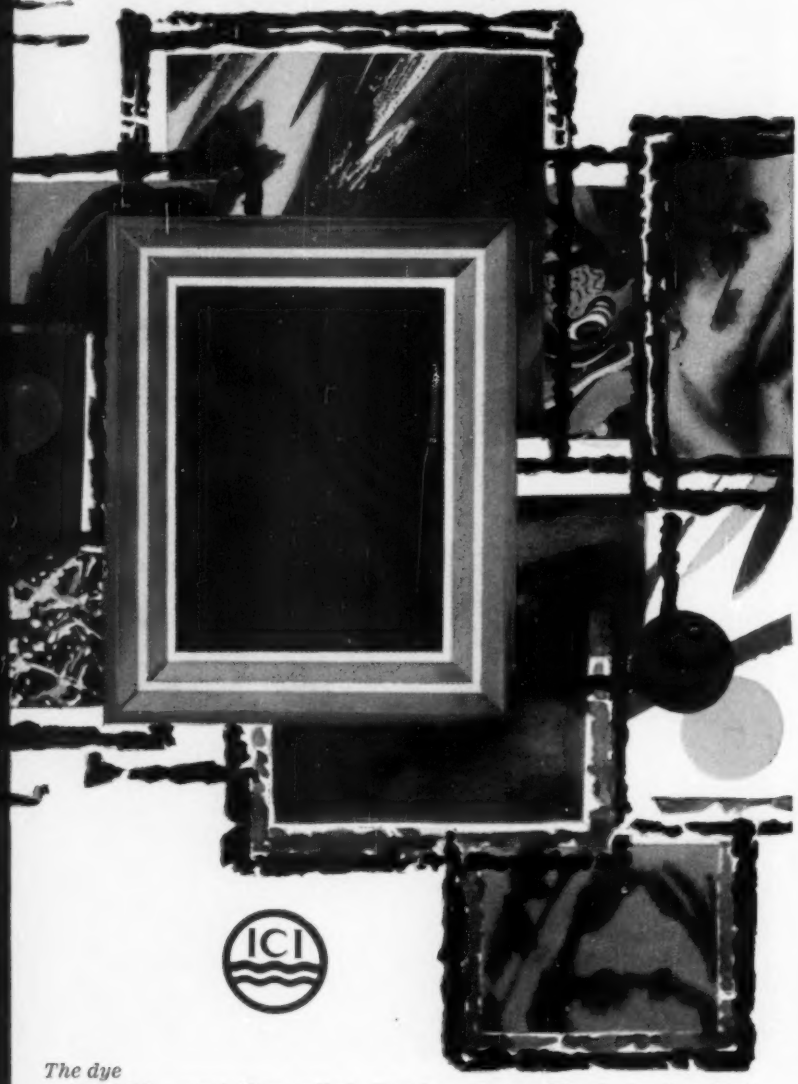

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Volume 77 Number 10 OCTOBER 1961 *Issued Monthly*

COMMUNICATIONS

The Formation of Polymers in Wool

L. J. WOLFRAM and J. B. SPEAKMAN

The preferential internal deposition of polymer, which is brought about by first impregnating wool with a solution of potassium persulphate in 0·1-N sulphuric acid, and then adding acrylonitrile in an atmosphere of nitrogen, is not due to the formation of free radicals by the action of persulphate on cystine cross-linkages. Owing to the affinity of wool for persulphate ions, their concentration inside the fibres greatly exceeds that in the surrounding medium, and free radicals are thus formed preferentially inside the fibres: $S_2O_8^{--} \rightarrow 2\ SO_4^{-}$·, leading to preferential internal deposition of polymer. As would be expected, therefore, polymer is readily formed in Fibrolane, but not in wool whose basic groups have been blocked by acetylation or treatment with 1-fluoro-2,4-dinitrobenzene, nor in wool which has been oxidised with peracetic acid.

Introduction

Polymer is formed preferentially inside wool fibres when they are treated with a solution containing potassium persulphate and methacrylic acid [1]. In order to explain this result it was suggested that polymerisation is initiated within the fibres by the free radicals which are formed by the action of persulphate on cystine cross-linkages—

$$\text{R–S–S–R} \rightarrow 2\text{R–S}\cdot$$
$$\text{R–S}\cdot + S_2O_8^{--} \rightarrow \text{R·S·O·SO}_2^{-} + \cdot SO_4^{-}$$
$$\text{R·S·O·SO}_2^{-} \rightarrow \text{R–S}\cdot + \cdot SO_4^{-}$$
$$\cdot SO_4^{-} + H_2O \rightarrow HSO_4^{-} + \cdot OH$$

Although the wool–persulphate system has since been used with other monomers [2], the validity of the above reaction scheme has never been examined, and the present investigation was undertaken to repair the omission. Acrylonitrile was chosen as the monomer because it is neutral and water-soluble, and polymerises readily.

Experimental

PREPARATION OF MATERIALS

Flannel

The all-wool flannel had the following construction—

Warp: 28·4s Yorkshire skeins, 32 ends/in.
Weft: 26·6s Yorkshire skeins, 34 picks/in.
Weave: plain Weight: 5·5 oz/yd²

Samples of the flannel were purified by extraction with ether and ethanol in a Soxhlet apparatus, followed by washing in running water. To remove traces of iron from the wool, it was immersed in a large volume of 0·01-N hydrochloric acid for 48 h at room temperature and then washed in several changes of distilled water until the pH of the solution in equilibrium with the wool rose to 5·0. The flannel was then removed, centrifuged, and conditioned at 65% r.h. and 22·2°C.

Worsted Fabric

The crossbred fabric, which had the following construction, was purified in the same way as the flannel.

Warp: 2/30s worsted, 50 ends/in.
Weft: 2/26s worsted, 30 picks/in.
Weave: plain Weight: 6·0 oz/yd²

Lincoln Wool

The fibres were purified in the same way as the all-wool flannel.

Fibrolane (3·2 den)

After being extracted for 6 h with ether in a Soxhlet apparatus, the fibres were freed from traces of iron by the procedure followed with the all-wool flannel.

Acrylonitrile

The B.D.H. reagent was re-distilled under atmospheric pressure and the fraction boiling at 78·0–78·5°C was collected.

Potassium Persulphate

The AnalaR reagent was used without further purification.

Cysteine and Cystine Hydrochlorides

The B.D.H. reagents were recrystallised before use.

Tyrosine, Methionine, and Lanthionine

The B.D.H. reagents were used without further purification.

PREPARATION OF MODIFIED FIBRES

Reduction

The wool was treated for 18 h at 35°C with a solution of thioglycollic acid (0·5 M), brought to pH 4·8 with sodium hydroxide, the liquor : wool ratio being 50 : 1. An alternative procedure was to treat the wool with sodium sulphide solution (0·042 M) for 20 min at 25°C, the liquor : wool ratio being 50 : 1. After either treatment the wool was washed in running water for 24 h, treated with 0·01-N hydrochloric acid for 12 h, brought to pH 5 with distilled water, centrifuged, and conditioned in a room maintained at 65% r.h. and 22·2°C.

Oxidation

The wool was treated with a 1·6% solution of peracetic acid for 24 h at 25°C, the liquor : fibre ratio being 100 : 1. After treatment, the fibres were washed in running water for 12 h, rinsed in several changes of distilled water, centrifuged, and conditioned.

Formation of Lanthionine

Samples of wool were boiled for 30 min in potassium cyanide solution (0·65%), using a liquor ratio of 40 : 1. The fibres were washed in running water, and then in several changes of distilled water before being centrifuged and conditioned.

Dinitrophenylation

The wool (1 g) was treated for 23 h at 40°C with a solution containing potassium bicarbonate (1 g), 1-fluoro-2,4-dinitrobenzene (1·75 g), distilled water (25 ml), and ethanol (50 ml). After being extracted for 3 h with ethanol in a Soxhlet apparatus, the wool was washed in running water overnight, rinsed in several changes of distilled water, centrifuged, and conditioned.

Acetylation

Dry wool (2 g) was treated for 50 min at 57°C with a solution containing acetic anhydride (7·8 g), glacial acetic acid (22·2 g), sulphuric acid (0·72 g), and dimethylaniline (0·4 g). After being washed in running water overnight, the wool was extracted with ethanol for 3 h in a Soxhlet apparatus, rinsed in several changes of distilled water, centrifuged, and conditioned.

ANALYTICAL METHODS

Determination of Acetyl Content

A sample of the acetylated wool (1 g conditioned weight) was heated with phosphoric acid (40 ml, 50%) and xylene (120 ml) until the wool dissolved, and the solution then distilled. The distillate was collected in an aqueous solution of potassium iodide, and the iodine liberated on addition of potassium iodate was estimated by adding excess of sodium thiosulphate and back-titrating with iodine. A blank experiment was carried out on the untreated wool.

Estimation of Potassium Persulphate

An aliquot (2 ml) of the solution was diluted to 30 ml with distilled water. After potassium iodide (4 g) had been added, the flask was stoppered and allowed to stand for 15 min. The solution was then acidified with a few drops of sulphuric acid (0·1 N) and the liberated iodine titrated with sodium thiosulphate.

Degree of Polymerisation

The molecular weight of the polymer formed under various conditions was obtained by determining the viscosity of a series of solutions, ranging in concentration from 0·05 to 0·3 g/100 ml, in dimethylformamide. After the limiting viscosity had been obtained by extrapolation, the molecular weight was calculated from Frind's equation [3].

POLYMERISATION PROCEDURE

After pretreatment, if necessary, the conditioned pattern (1·2 g) was introduced into a 250-ml three-necked, round-bottomed flask, which was immersed in a thermostat at the desired temperature (±0·1°C). A solution of potassium persulphate in 0·1-N sulphuric acid (100 ml) was then added and, after the pressure had been reduced to about 15 mm of mercury for 3 min, oxygen-free nitrogen was admitted and allowed to bubble through the solution for 15 min. At this stage acrylonitrile (5 ml) was added from a dropping funnel, the contents of the flask being agitated until the solution became homogeneous.

At the end of the reaction period, the pattern was removed from the flask, washed in running water for at least 2 h, centrifuged, conditioned, and weighed. All the experiments were carried out in duplicate, and the results given in subsequent tables are mean values for the gain in weight, based on the weights of the conditioned, untreated patterns.

Results

EFFECT OF AMINO ACIDS ON POLYMERISATION OF ACRYLONITRILE

Of all the amino acids present in keratin, tyrosine and those that contain sulphur are the ones that are most likely to promote decomposition of persulphate into radicals. Experiments with these amino acids in $1{\cdot}35 \times 10^{-3}$ M concentration were therefore carried out at 25°C, the extent of conversion into polymer being determined by weighing the amount of polymer precipitated after a suitable time of reaction in an atmosphere of nitrogen. The results are given in Table I.

As none of the amino acids except cysteine is effective in shortening the induction period and increasing the rate of polymerisation, it was decided to examine the persulphate–cysteine initiator system more closely before making experiments on wool.

POLYMERISATION OF ACRYLONITRILE BY THE PERSULPHATE–CYSTEINE SYSTEM

In 1946 Bacon [4] discovered that an acidic medium favours the polymerisation of acrylonitrile by persulphate, especially if the system is activated by a reducing agent. In order to discover the optimum conditions for the persulphate–cysteine system, polymerisation experiments were therefore carried out in solutions containing various

TABLE I

Concentration of acrylonitrile: $7{\cdot}22 \times 10^{-1}$ M
Concentration of persulphate: $11{\cdot}2 \times 10^{-3}$ M

Medium	Amino acid	Induction period (min)	Percentage conversion after 2 h	Percentage conversion after 4 h
Water	None*	16	2·8	5·8
	Cystine hydrochloride	17	2·5	5·6
	Cysteine hydrochloride	2	37·5	—
	Methionine*	18	2·4	5·3
	Lanthionine*	20	2·0	4·9
	Tyrosine*	25	1·9	4·8
0·1-N H_2SO_4	None	20	—	3·9
	Cystine hydrochloride	18	—	3·8
	Cysteine hydrochloride	21	—	6·6
	Methionine	19	—	2·4
	Lanthionine	22	—	3·1
	Tyrosine	25	—	2·1

* An amount of hydrochloric acid equivalent to that combined with cystine and cysteine was also added in these cases.

concentrations of sulphuric acid. Cysteine hydrochloride was dissolved in distilled water and neutralised with sodium hydroxide before being added to the reaction medium, a corresponding amount of sodium chloride being used in the blank experiments with persulphate alone. The results are summarised in Table II.

TABLE II

Concentration of acrylonitrile: $7{\cdot}22 \times 10^{-1}$ M
Concentration of persulphate: $11{\cdot}2 \times 10^{-3}$ M
Concentration of cysteine: $7{\cdot}0 \times 10^{-3}$ M
Temperature: 25°C

Initial pH	Rate of polymerisation (%/min) × 10^3	
	Persulphate alone	Persulphate + cysteine
6·5	3·8	7·9
5·0	3·9	65·5
4·0	—	66·5
3·0	2·5	70·0
2·1	2·0	49·4
1·2	1·6	2·5

Low concentrations of sulphuric acid (pH 3 to 5) have a striking effect in promoting polymerisation by the persulphate–cysteine system, and it seems possible that the low rates of polymerisation at pH 1·2, in presence or absence of cysteine, are due to the fact that heterogeneous decomposition of persulphate, which does not proceed through the free-radical species, is favoured by low values of pH.

The dependence of the rate of polymerisation on the concentration of cysteine was also examined under the above conditions at pH 3; the results are given in Table III.

TABLE III

Concentration of cysteine (10^{-3} M)	Rate of polymerisation (%/min) × 10^3	Degree of polymerisation (DP)
None	2·5	6500
0·35	12·7	3570
0·70	39·4	2260
1·35	53·5	1890
3·50	67·1	1265
7·00	70·0	905

Although the rate of polymerisation increases with increasing concentration of cysteine, the change is slight when the concentration is relatively high. It is, however, accompanied by a decrease in the degree of polymerisation, probably because the cysteine acts as a chain-transfer agent. These results are of some importance in relation to the role of thiols in emulsion polymerisation [5].

POLYMERISATION OF ACRYLONITRILE INSIDE UNTREATED WOOL FIBRES

The influence of various factors on the formation of polymer inside wool fibres was examined in order to define the optimum conditions of treatment.

Effect of pH

The worsted fabric was used and the results obtained with various concentrations of sulphuric acid in the reaction medium are given in Table IV.

TABLE IV

Concentration of acrylonitrile: $7{\cdot}22 \times 10^{-1}$ M
Concentration of persulphate: $11{\cdot}2 \times 10^{-3}$ M
Treated for 2 h at 25°C

Initial pH	Increase in weight (%)
5·5	1·1
4·2	5·8
3·1	12·2
2·1	23·9
1·2	22·9

During the course of these experiments it was noticed that the period of induction before polymer was formed in the *solution* varied from 15–25 min in dilute solutions of sulphuric acid to 60 min or more at pH 2·1 and 1·2, the difference being much greater than in the corresponding experiments (Table II) with acrylonitrile in absence of wool.

Comparison of the results of Table IV with those of Table II shows that the ratio of the amount of polymer formed in the wool to the total amount of polymer produced will be greatest at pH 1·2.

Concentration of Potassium Persulphate

Worsted fabric was again used, polymerisation being allowed to proceed for 2 h at 25°C in sulphuric acid at an initial pH of 1·2. The results are given in Table V.

TABLE V

Concentration of acrylonitrile: $7{\cdot}22\times10^{-1}$ M

Concentration of persulphate (10^{-3} M)	Increase of weight (%)
3·7	12·2
7·5	20·2
11·2	22·9
14·8	23·2
22·4	23·0

It was in the light of these results that a persulphate concentration of $11{\cdot}2\times10^{-3}$ M was chosen for use in most of the experiments.

Concentration of Acrylonitrile

The results of experiments with the worsted fabric and different concentrations of acrylonitrile are given in Table VI.

TABLE VI

Concentration of persulphate: $11{\cdot}2\times10^{-3}$ M
Treated for 2 h at 25°C Initial pH: 1·2

Concentration of acrylonitrile ($\times10^{-1}$ M)	Increase in weight (%)
3·70	11·8
7·22	22·9
10·5	30·5

The amount of polymer formed in the wool is at first proportional to the concentration of acrylonitrile. On the basis of these results a concentration of $7{\cdot}22\times10^{-1}$ M was adopted in most of the experiments.

Temperature

The results of the experiments with worsted fabric at different temperatures and pH 3·1 are given in Table VII.

When the logarithms of the initial slopes of the curves showing the rate of deposition of polymer are plotted against $1/T$, a straight line is obtained. The apparent energy of activation, calculated from the slope of the line, is 7·9 kcal., in good agreement with the value of 7·1 kcal. obtained by Valentine [6] for the polymerisation of acrylonitrile in wool by means of the ferrous ion–hydrogen peroxide system.

A second set of experiments was carried out with flannel under the same conditions as above, except that the initial pH of the medium was 1·2. The results are given in Table VIII.

In this case the activation energy was found to be 9·2 kcal., the value being higher than for crossbred worsted fabric because of the lower rate of diffusion of monomer through the more compact structure of the flannel.

Microscopic examination of fibres from the fabrics used in the preceding experiments showed that there was no external deposit of polymer. When conditions are not such as to promote preferential internal polymerisation, a superficial deposit can be detected with as little as 3% of polymer on the fibres. Even when the amount of polyacrylonitrile deposited in Lincoln wool was as high as 49·8%, exhaustive extraction with dimethylformamide, in which the polymer is readily soluble, reduced the polymer content of the wool only to 46·8%.

POLYMERISATION OF ACRYLONITRILE INSIDE MODIFIED WOOL FIBRES

Lanthionine-containing Wool

The results obtained on flannel treated with potassium cyanide are given in Table IX. From these data the activation energy of the reaction was found to be 16·2 kcal.

Reduced Wool

Polymerisation experiments were carried out with samples of Lincoln wool which had been

TABLE VII

Concentration of acrylonitrile: $7{\cdot}22\times10^{-1}$ M
Concentration of persulphate: $11{\cdot}2\times10^{-3}$ M

Temperature	Percentage increase in weight after: (min)						Initial slope
(°C)	30	60	90	120	180	210	(%/min)
25·0	—	6·7	—	12·2	—	17·1	0·117
37·0	6·1	10·2	14·0	17·4	20·5	—	0·203
53·5	11·3	16·6	—	21·3	—	23·7	0·377

TABLE VIII

Concentration of acrylonitrile: $7{\cdot}22\times10^{-1}$ M
Concentration of persulphate: $11{\cdot}2\times10^{-3}$ M

Temperature	Percentage increase in weight after: (min)					Initial slope
(°C)	30	60	90	120	180	(%/min)
25	—	6·2	9·2	11·9	15·5	0·113
45	8·4	12·5	16·1	17·9	—	0·306

TABLE IX

Concentration of acrylonitrile: $7{\cdot}22\times10^{-1}$ M
Concentration of persulphate: $11{\cdot}2\times10^{-3}$ M
Initial pH: 1·2

Temperature	Percentage increase in weight after: (min)						Initial slope
(°C)	25	40	60	90	120	150	(%/min)
25	—	8·7	13·1	19·5	25·0	28·8	0·218
45	22·2	28·7	—	39·2	—	49·1	1·20

reduced by the methods described on p. 478; the results are given in Table X.

TABLE X

Concentration of acrylonitrile: $7{\cdot}22\times10^{-1}$ M
Concentration of persulphate: $11{\cdot}2\times10^{-3}$ M
Conditions: 2 h at 25°C

Reducing agent	Polymerisation medium	Increase in weight (%)
Thioglycollic acid	Water	8·6
	0·1-N H_2SO_4	36·0
Sodium sulphide	Water	16·2
	0·1-N H_2SO_4	35·1
None	Water	0·2
	0·1-N H_2SO_4	27·5

Acetylated and Dinitrophenylated Wools

Polymerisation experiments were carried out with samples of crossbred fabric which had been acetylated or dinitrophenylated under the conditions previously described. The results are given in Table XI.

TABLE XI

Concentration of acrylonitrile: $7{\cdot}22\times10^{-1}$ M
Concentration of persulphate: $11{\cdot}2\times10^{-3}$ M
Conditions: 2 h at 25°C in 0·1-N H_2SO_4

Wool	Increase in weight (%)
Untreated	22·9
Acetylated	−0·1
Dinitrophenylated	0·3

The untreated wool had an apparent acetyl content of 0·48%, while that of the acetylated wool was 3·21%.

Oxidised Wool

The results of polymerisation experiments with crossbred fabric which had been oxidised with peracetic acid are given in Table XII.

TABLE XII

Concentration of acrylonitrile: $7{\cdot}22\times10^{-1}$ M
Concentration of persulphate: $11{\cdot}2\times10^{-3}$ M
Conditions: 0·1-N H_2SO_4 at 25°C

Time of treatment (h)	Increase in weight (%)
2	−0·9
4	−1·3
6	−1·5

Similar experiments with crossbred fabric which had been oxidised by treatment with a solution of potassium persulphate ($11{\cdot}2\times10^{-3}$ M) in sulphuric acid (0·1 N) for 24 h at 25°C gave the results shown in Table XIII, the time of polymerisation being 2 h.

POLYMERISATION OF ACRYLONITRILE IN FIBROLANE

The results of experiments on the polymerisation of acrylonitrile in Fibrolane are given in Table XIV.

TABLE XIV

Concentration of acrylonitrile: $7{\cdot}22\times10^{-1}$ M
Concentration of persulphate: $11{\cdot}2\times10^{-3}$ M
Medium: 0·1-N H_2SO_4

Temperature	Percentage increase in weight after: (min)						Initial slope
(°C)	22	45	60	90	120	180	(%/min)
25	—	—	16·6	24·5	32·5	37·5	0·27
45	19·1	32·0	38·5	—	53·2	—	0·95

TABLE XIII

Time of oxidation (h)	Increase in weight (%)
0	22·9
12	4·5
24	3·1

From these results the activation energy was found to be 11·9 kcal. It is interesting that the acidic solutions of potassium persulphate, in striking contrast to the ferrous ion–hydrogen peroxide system [7], should be so successful in promoting preferential internal polymerisation of acrylonitrile.

ABSORPTION OF PERSULPHATE BY UNTREATED AND MODIFIED WOOLS AND FIBROLANE

Untreated Wool

The rates of absorption of persulphate by the worsted fabric from solutions of various concentrations in 0·1-N sulphuric acid at 25°C are shown by the data of Table XV, the liquor : wool ratio being 100 : 1.

TABLE XV

Concentration of persulphate ($\times10^{-3}$ M)	Amount of persulphate absorbed (m equiv./g dry wool) in: (min)					
	15	30	60	120	180	1440
3·4	—	0·13	0·19	0·27	0·33	—
4·6	—	0·17	0·27	0·41	0·48	—
8·7	—	0·33	0·47	0·62	0·70	—
11·5	0·25	0·37	0·53	0·65	—	—
15·6	—	0·40	0·56	0·68	0·75	—
22·4	—	0·43	0·60	0·71	0·78	0·88

As the amounts of persulphate absorbed were calculated from the change in concentration of the solution after contact with the wool for the specified time, the above values include the amounts of persulphate lost by reaction with the wool. It was, however, found that most of the persulphate could be recovered unchanged by rinsing the wool in water.

The influence of pH on the absorption of persulphate at 25°C is shown by the results of Table XVI, the time of treatment being 30 min, and the initial concentration of persulphate $11{\cdot}5\times10^{-3}$ M.

TABLE XVI

Concentration of sulphuric acid (N)	Amount of persulphate absorbed (m equiv./g dry wool)
nil	0·06
0·001	0·19
0·010	0·39
0·100	0·37

Oxidised Wool

The rate of uptake of persulphate by peracetic-acid-oxidised wool from a solution of persulphate

($11{\cdot}2\times10^{-3}$ M) in sulphuric acid (0·1 N) at 25°C is shown by the data of Table XVII. A liquor ratio of 100 : 1 was used.

TABLE XVII

Time (min)	Amount of persulphate absorbed (m equiv./g dry wool)
15	0·00
30	0·05
60	0·08
240	0·10

Lanthionine-containing Wool

Similar experiments to the preceding on flannel treated with potassium cyanide gave the results summarised in Table XVIII.

TABLE XVIII

Time (min)	Amount of persulphate absorbed (m equiv./g dry wool)
15	0·38
30	0·66
60	0·78
127	0·94
182	1·09

Acetylated and Dinitrophenylated Wools

No persulphate was absorbed by the acetylated wool in 60 min, and even the dinitrophenylated wool absorbed no more than 0·06 m equiv./g in 60 min under the above conditions.

Fibrolane

The experiments were carried out under the same conditions as with wool; the results are given in Table XIX.

TABLE XIX

Time (min)	Amount of persulphate absorbed (m equiv./g dry fibre)
11	0·44
20	0·53
30	0·60
45	0·63
60	0·65
120	0·66

Discussion

Of the amino acids which are likely to promote the formation of free radicals by reaction with potassium persulphate, only cysteine was found to promote the polymerisation of acrylonitrile by persulphate. With the persulphate–cysteine system, polymerisation is most rapid at pH 3 and the rate increases with increasing concentration of cysteine. Since untreated wool contains little cysteine, and since the persulphate–cystine system does not promote polymerisation, it is at first sight surprising that polyacrylonitrile is formed so rapidly inside wool fibres when they are treated with a solution of persulphate and acrylonitrile (Tables IV, V, and VI). It seems unlikely that the small amount of cysteine in the wool is responsible for this result, because the amount of polymer deposited at pH 1·2 is considerable (Table IV), whereas cysteine at the same pH has a negligible effect in promoting polymerisation (Table II). On the other hand, greater amounts of polymer are formed in reduced wool than in untreated wool when solutions of persulphate in both water and sulphuric acid are used (Table X). Similarly, the possibility that the cystine of wool may promote polymerisation by persulphate is not eliminated by the experiments with cyanide-treated wool (Table IX), because the conversion of cystine into lanthionine cross-linkages is accompanied by an increase in the cysteine content of the wool. Conclusive proof that neither cysteine nor cystine plays an essential role in promoting preferential internal polymerisation of acrylonitrile is, however, provided by the fact that polymer is readily formed in Fibrolane (Table XIV), which contains only a trace of cystine. How, then, is polyacrylonitrile formed inside the fibres?

The answer to this enquiry is given by the failure of persulphate to promote preferential polymerisation of acrylonitrile in acetylated and dinitrophenylated wools (Table XI). Unlike untreated wool (Table XV) and Fibrolane (Table XIX), both of which combine with persulphate preferentially at pH 1·2, the treated wools have little or no affinity for persulphate. It seems clear, therefore, that preferential formation of polymer inside untreated wool and Fibrolane is due to the fact that the concentration of persulphate ions inside the fibres at pH 1·2 is greatly in excess of the concentration in the surrounding medium. Since the rate of formation of ion radicals from the persulphate ion is of first order with respect to the concentration of persulphate—

$$S_2O_8^{=}\rightarrow 2\ SO_4^{-\cdot}$$

it is obvious that the formation of free radicals and, therefore, of polymer, will take place preferentially inside the fibres. This view is supported by the fact that little or no polymer is deposited in oxidised wool (Tables XII and XIII) under conditions which give large deposits in untreated wool, in keeping with the fact that the oxidised wool has little affinity for persulphate at pH 1·2 (Table XVII). The lower affinity for persulphate of wool oxidised with peracetic acid is, of course, due to the abundance of cysteic acid side-chains in the fibres. Conversely, polymer is readily formed inside cyanide-treated fibres, in keeping with the fact that the affinity for persulphate is unimpaired (Table XVIII).

* * *

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Shrinkproofing of Wool with Reducing Agents

A. J. FARNWORTH

Wool can be shrinkproofed by treatment with ethanolic or aqueous solutions of ammonium thioglycollate or of mixtures of thioglycollic acid and organic bases. Preliminary extraction of the fabric with ethanol greatly increases the effectiveness of aqueous solutions of ammonium thioglycollate. Extracted fabric can also be shrinkproofed by treatment with aqueous solutions of sodium bisulphite, which are without effect on unextracted fabric. However, the shrink resistance, which withstands washing in hot water, is largely lost on washing in soap solution.

Shrinkproofing with reducing agents can be achieved under conditions where reaction very probably occurs only with disulphide bonds. Re-building of disulphide, or formation of new covalent, bonds after the shrinkproofing reaction does not destroy the unshrinkability. Treatment with aqueous solutions of reducing agents leads to unshrinkability without the reaction being confined to the surface of the fibre, and without affecting the difference between the with-scale and anti-scale coefficients of friction. The effect cannot be explained in terms of the conventional theories of shrinkproofing.

Introduction

Although there is some evidence to suggest that shrinkproofing of wool requires only the breakdown of disulphide bonds at, or near, the surface of the fibres [1,2], no satisfactory method has been developed for shrinkproofing directly with reducing agents which are specific reagents for disulphide bonds. Methods involving the use of reducing agents require either a treatment subsequent to reduction—e.g., methylation [3], or treatment with enzymes [4], chloromethyl ether [5], organic thiosulphates [6], or resins [7]—or a preliminary oxidation [8]. Alexander *et al.* [9] showed that oxidation of disulphide bonds alone was insufficient to give unshrinkability, and suggested that the wool must be attacked simultaneously at the tyrosine residue. It has also been shown that a combination of disulphide oxidation and peptide hydrolysis is required to shrinkproof wool with sulphuryl chloride [10]. However, oxidation by this reagent probably would not be confined to disulphide bonds.

In this paper it is shown that wool can be shrinkproofed with reducing agents under conditions where reaction is almost certainly confined to disulphide bonds. The particular reagents studied were thioglycollates in both ethanolic and aqueous solution, and bisulphites in aqueous solution. It is doubtful whether the methods described would have practical application.

Materials and Methods

Fabric—The fabric used was a scoured plain-weave cloth made from Merino 64s wool and with the following construction—

Warp	30 tex × 2; 32 ends/in.
Weft	30 tex × 2; 30 picks/in.
Weight	4·5 oz/yd².

Felting Shrinkage—Fabric samples 6-in. square were relaxed in a solution containing 0·2% of anionic detergent (Teepol 610—Shell Chemicals) and 0·2% sodium bicarbonate. The area was measured whilst the fabrics were still wet, and the fabrics were then washed, together with non-felting ballast to a total load of 1 kg, in a domestic tumbler-type washing machine containing water at 40°C and pH 7·5, until an untreated control pattern showed an area shrinkage of 40%.

Alkali Solubility—This was determined by the method of Harris and Smith [11].

Acid Solubility—This was determined by the method of Zahn and Würz [12].

Yarn Strength—Yarn was separated from the fabric samples and its breaking strength determined on a Scott tensile tester.

Bursting Strength—This was determined with a standard Mullen-type bursting strength tester on fabrics conditioned at 65% r.h. and 20°C, and also after wetting in 0·2% (v/v) Teepol solution.

Abrasion Resistance—Loss in weight was determined after rubbing the fabric in a Martindale tester against stainless steel gauze (40 mesh) for 1000 cycles, using a 14½-oz weight on the pattern holder.

Friction Measurement—Coefficients of friction of Lincoln 36s wool fibres were measured by the capstan method of Lipson and Howard [13]. Measurements were made on air-dry fibres and also when fibre and keratin rod were lubricated with 0·05-M sodium borate solution containing 0·02% (v/v) Teepol.

Results

EFFECT OF COMBINING DISULPHIDE REDUCTION AND PEPTIDE HYDROLYSIS

Fabrics were treated successively at 35°C with 2-N HCl for 18 h and 0·5-M sodium thioglycollate at pH 5 for 2 h. Although each individual treatment did not affect the felting shrinkage to any great extent, successive treatments reduced the area shrinkage from 40% to 7%. Although the fabrics were damaged, the results illustrate that breakdown of disulphide bonds and of peptide linkages can lead to unshrinkability.

TREATMENT WITH ETHANOLIC SOLUTIONS OF THIOGLYCOLLIC ACID AND ORGANIC BASES

Bradbury [14] had found that a high degree of unshrinkability resulted from treatment of wool with solutions of hydrazine in ethanol, and it was decided to investigate the effect of combining this treatment with simultaneous reduction of disulphide bonds. Fabrics were treated with ethanolic solutions of hydrazine hydrate and thioglycollic acid at 35°C for 30 min at a liquor ratio of 20 : 1 and then rinsed thoroughly in running water. Table I shows that excellent shrink resistance can be obtained with a mixture of these reagents under conditions where each individually is not nearly so effective.

TABLE I

Treatment with Ethanolic Solutions of Thioglycollic Acid and Hydrazine Hydrate

Concentration of thioglycollic acid % (v/v)	Concentration of hydrazine hydrate % (v/v)	Area shrinkage (%)
0	0	40
0	5	13
5	0	32
0·5	2	3
1	1	8
1	3	2
1	4	0
2	2	5
2	4	0
3	5	0

Further investigations showed that other organic bases were effective when used in conjunction with thioglycollic acid. Monoethanolamine, dimethylamine, and triethanolamine all produced marked decreases in felting shrinkage, but hydrazine and ethylenediamine were the most effective. Table II shows the felting shrinkage and various properties of the treated fabrics after treatment with ethanolic solutions of thioglycollic acid and either hydrazine or ethylenediamine under a variety of conditions.

It is seen that unshrinkability can be obtained under mild conditions of treatment with very little chemical degradation. As in other antishrink treatments using ethanolic solutions, yarn strength is actually increased. However, the addition of water, whilst not affecting the shrink resistance, leads to marked chemical degradation.

TABLE II

Treatment with Ethanolic Solutions of Thioglycollic Acid and Hydrazine Hydrate or Ethylenediamine

Concentration of thioglycollic acid % (v/v)	Concentration of hydrazine hydrate % (v/v)	Concentration of ethylene-diamine % (v/v)	Liquor ratio 10 : 1 Conditions Time (min)	Temp. (°C)	Area shrinkage (%)	Acid solubility (%)	Alkali solubility (%)	Weight loss on abrasion (mg)	Yarn strength (arbitrary units)
0	0	0	—	—	40	5·5	13·1	8·4	529
1	2	—	240	25	0	8·4	15·9	12·4	577
1	2	—	30	35	3	6·6	13·5	7·0	590
1	2	—	30	50	2	8·5	19·1	7·2	594
1	3	—	30	35	3	6·1	12·6	7·4	579
2	4	—	30	35	1	8·9	15·3	6·7	596
5	5	—	30	35	1	10·7	20·9	9·1	642
1*	3	—	30	35	4	12·9	30·4	10·8	530
5	5	—	Padded to 100% uptake and dried at 105°C		1	10·7	16·6	9·7	584
1	—	5	30	35	4	6·1	11·4	6·3	587
2	—	5	30	35	3	6·5	11·4	4·5	589
5	—	5	30	35	1	6·5	12·5	7·2	584
5	—	5	Padded to 100% uptake and dried at 105°C		8	7·9	14·3	7·5	563

* 20% of water (by vol.) added.

TABLE III

Treatment with Ethanolic Solutions of Ammonium Thioglycollate

Concentration of thioglycollic acid % (v/v)	Concentration of ammonia (sp. gr. 0·880) % (v/v)	Liquor ratio 10 : 1 Conditions Time (min)	Temp. (°C)	Area shrinkage (%)	Acid solubility (%)
0	0	—	—	40	5·5
1	1	30	25	25	—
1	6	30	25	7	—
1	6	30	35	3	6·9
0·25	6	15	40	15	—
0·25	6	30	40	6	8·0
0·5	0·5	15	40	17	—
0·5	6	15	40	7	—
0·5	6	30	40	4	8·0
1	1	15	40	22	—
1	6	30	40	2	10·9
0·25	6	15	55	15	—
0·25	6	30	55	7	8·3
0·5	1	30	55	14	—
0·5	6	30	55	4	10·0
1	6	15	55	1	13·6

TABLE IV

Treatment with Boiling Ethanolic Solutions of Ammonium Thioglycollate

Liquor ratio 10 : 1 Temperature 80°C

Concentration of thioglycollic acid % (v/v)	Concentration of ammonia (sp. gr. 0·880) % (v/v)	Time (s)	Area shrinkage (%)	Weight loss on abrasion (mg)	Yarn strength (arbitrary units)
0	0	—	40	8·4	529
0·25	6	60	20	—	—
0·5	6	60	6	—	—
1	6	30	11	8·5	516
1	6	60	4	5·7	550
1	6	120	3	7·0	582
2	6	30	4	6·0	552
2	6	60	0	6·6	575

TREATMENT WITH ETHANOLIC SOLUTIONS OF AMMONIUM THIOGLYCOLLATE

As it seemed unlikely that the hydrazine and various amines were functioning other than as bases, the possibility of replacing them by ammonia was investigated. Conditions of temperature, concentration, time, etc. were varied over a wide range; Tables III and IV provide a selection of the results obtained.

The main conclusions drawn from these results were that—

(*a*) relatively low concentrations of ammonium thioglycollate (0·25–0·5%), in the presence of excess of ammonia, can impart good shrink resistance under mild conditions without appreciable degradation

(*b*) boiling solutions give good shrink resistance in 30–60 s without any apparent adverse effect on the fabric.

Further investigations showed that other thiols could be used instead of thioglycollic acid, and the regain of the wool is unimportant, dry wool being no less effectively treated than wool at 16% regain. It was also found that a combination of thioglycollic acid, ammonia, and hydrazine hydrate gave very good shrink resistance at low concentrations of reagents (e.g. 0·25% (v/v) thioglycollic acid, 1% (v/v) hydrazine hydrate, 0·25% (v/v) ammonia at 40°C for 1 h; 0·25% thioglycollic acid, 0·5% hydrazine, 0·25% ammonia at 55°C for 30 min, without any adverse effect on wet or dry bursting strength.

TABLE V

Treatment with Aqueous Solutions of Thioglycollic Acid and Hydrazine Hydrate

Liquor ratio 10 : 1 Duration of treatment 30 min

Concentration of thioglycollic acid % (v/v)	Concentration of hydrazine hydrate % (v/v)	Area shrinkage (%)	Bursting strength (lb/in²) Dry	Wet
0	0	40	113	83
Treatments at 40°C				
0	5·0	36	—	—
5·0	0	40	—	—
0·25	5·0	20	—	—
0·5	5·0	12	—	—
1·0	5·0	12	102	71
2·0	3·0	10	102	69
2·0	4·0	6	100	70
2·0	5·0	5	101	66
Treatments at 55°C				
0	5·0	23	—	—
5·0	0	32	—	—
0·25	5·0	16	—	—
0·5	3·0	10	104	71
1·0	3·0	9	101	70
1·0	5·0	6	101	70
2·0	3·0	6	94	68
5·0	5·0	4	99	62

TREATMENT OF UNEXTRACTED FABRIC WITH AQUEOUS SOLUTIONS OF THIOGLYCOLLIC ACID AND HYDRAZINE

Attempts to shrinkproof wool by treatment with aqueous solutions of ammonium thioglycollate showed that, with fairly severe treatments, the area shrinkage could be reduced from 40% to about 20%. However, as can be seen from Table V, much better results were obtained by using aqueous solutions of thioglycollic acid and hydrazine.

TREATMENT OF ETHANOL-EXTRACTED FABRIC WITH AQUEOUS OR ETHANOLIC SOLUTIONS OF AMMONIUM THIOGLYCOLLATE

Bradbury [14] had observed that ethanol-extracted fabrics behaved differently from unextracted fabrics when subsequently treated with aqueous solutions of hydrazine. In view of the large difference between the effectiveness of ethanolic and aqueous solutions of thioglycollates under the same conditions, it was decided to investigate the effect of ethanol extraction prior to treatment with aqueous solutions of thioglycollates.

Fabric samples were extracted with ethanol for 4 h in a Soxhlet apparatus, and then rinsed in running water. They were next conditioned at 65% r.h. and 20°C, and treated with aqueous or ethanolic solutions of ammonium thioglycollate under a variety of conditions. Unextracted fabrics were similarly treated for comparison. The results obtained are shown in Table VI.

It is seen that extraction with ethanol prior to treatment with aqueous thioglycollate solutions greatly enhances the shrinkproofing effect. Shrinkproofing can be obtained under conditions which have very little effect on unextracted fabric. Preliminary extraction enables the same degree of shrinkproofing to be obtained with aqueous solutions as with ethanolic solutions. In contrast, previous ethanol extraction has, in general, little effect when ethanolic solutions of thioglycollate are used, as shown by the results in Table VI and many other experiments.

TABLE VI

Treatment with Aqueous or Ethanolic Solutions of Ammonium Thioglycollate

Liquor ratio 10 : 1

Concentration of thioglycollic acid % (v/v)	Concentration of ammonia (sp. gr. 0·880) % (v/v)	Time (min)	Temp. (°C)	Area shrinkage (%) after treatment in *aqueous solution* unextracted fabric	*aqueous solution* extracted fabric	*ethanolic solution* unextracted fabric	*ethanolic solution* extracted fabric
0	0	—	—	40	40	40	40
0·1	0·1	120	30	33	17	29	25
0·1	3·0	60	40	27	10	—	—
0·1	3·0	30	55	28	6	—	—
0·1	6·0	15	55	30	6	—	—
0·25	0·25	15	55	36	20	30	28
0·25	3·0	60	40	25	3	—	—
0·25	3·0	15	55	28	0	—	—
0·25	6·0	15	40	26	8	12	5
0·25	6·0	15	55	23	5	9	7
0·5	0·5	60	40	26	10	24	13
0·5	6·0	15	40	27	8	12	12
0·5	6·0	15	55	26	7	8	10
1·0	1·0	60	40	30	8	20	8
1·0	6·0	120	30	26	7	3	1
1·0	6·0	15	55	25	4	0	0

TABLE VII

Properties of Fabrics after Treatment with Solutions of Ammonium Thioglycollate

Liquor ratio 10 : 1

	Control		Conditions of treatment: 0·25% thioglycollic acid, 6% ammonia, 40°C, 60 min		1% thioglycollic acid, 6% ammonia, 30°C, 120 min		1% thioglycollic acid, 6% ammonia, 55°C, 15 min	
	extracted fabric	unextracted fabric	aqueous soln. extracted fabric	ethanolic soln. unextracted fabric	aqueous soln. extracted fabric	ethanolic soln. unextracted fabric	aqueous soln. extracted fabric	ethanolic soln. unextracted fabric
Area shrinkage (%)	40	40	3	9	0	3	1	0
Bursting strength (dry) (lb/in²)	108	115	104	—	101	112	96	102
Bursting strength (wet) (lb/in²)	82	86	75	—	65	84	68	77
Alkali solubility (%)	11·0	13·5	15·5	11·0	45·8	18·5	33·7	17·2
Acid solubility (%)	8·3	8·3	11·8	8·0	29·6	12·3	20·0	12·2

The results in Table VII show that the effect on some of the properties of the wool is greater for aqueous treatment of extracted fabric than for ethanolic treatment of unextracted fabric under the same conditions.

TREATMENT OF ETHANOL-EXTRACTED FABRIC WITH AQUEOUS SOLUTIONS OF SULPHITES

Many unsuccessful attempts have been made to shrinkproof wool with aqueous solutions of sulphites. However, in view of the greatly enhanced effectiveness of thioglycollate treatments on ethanol-extracted wool, it was decided to investigate the effect of preliminary extraction on treatments with sulphites.

Tables VIII and IX and Fig. 1 show the effect of varying the conditions of treatment of ethanol-extracted wool with sodium sulphite or bisulphite. In Table VIII, results obtained in the treatment of unextracted fabric are included for comparison.

It is seen that shrink resistance can be imparted to extracted fabric with sulphites under conditions which are without effect on unextracted fabric.

TABLE VIII

Treatment with Aqueous Solutions of Sodium Bisulphite

Liquor ratio 10 : 1 pH 5·5 40°C for 1 h

Concentration of sodium bisulphite % (w/v)	Area shrinkage (%) Unextracted fabric	Extracted fabric
0	40	40
0·05	34	19
0·1	38	20
0·5	36	16
1·0	35	11
2·0	38	7
5·0	38	5

TABLE IX

Treatment of Ethanol-extracted Wool Fabric with Aqueous Solutions of Sodium Bisulphite

Liquor ratio 10 : 1 5% sodium bisulphite pH 5·5

Temperature (°C)	Time (min)	Area shrinkage (%)
Control	—	40
25	15	36
25	120	20
40	15	22
40	30	11
40	60	5
55	5	25
55	15	10
55	60	5

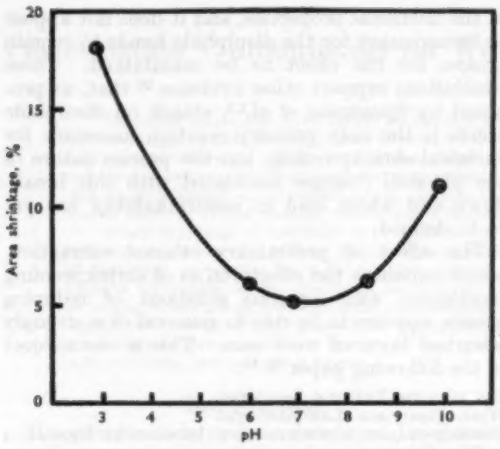

FIG. 1—Effect of pH on shrinkproofing with aqueous solutions of sulphites

This shrink resistance, which withstands machine washing in hot water, is largely lost on washing in soap solution [15].

EFFECT OF AFTERTREATMENT OF THE REDUCED WOOL

Fabrics which had been shrinkproofed with ethanolic solutions of ammonium thioglycollate (unextracted fabric) or with aqueous solutions of sodium bisulphite (extracted fabric) were (*a*) rinsed in running water for 24 h to re-form some of the disulphide bonds of the bisulphite-treated wool [16]; (*b*) methylated with methyl iodide to prevent the thiol groups being re-oxidised [17]; (*c*) treated with ethylene dibromide to cross-link the thiol groups through ethylene bridges [17]; (*d*) oxidised with hydrogen peroxide to re-form some disulphide bonds [18]. The results given in Table X show that none of these aftertreatments has any significant effect, with the possible exception of prolonged water rinsing of bisulphite-treated wool.

TABLE X

Aftertreatment of Wool Shrinkproofed with Reducing Agents

Aftertreatment	Area shrinkage %	
	Thioglycollate treatment*	Bisulphite treatment†
Control	40	40
None	0	6
Rinsed in running water for 24 h	2	11
Methylation	1	4
Cross-linking with ethylene dibromide	0	5
Oxidation with hydrogen peroxide	1	3

* Thioglycollate treatment—1% (v/v) thioglycollic acid, 6% (v/v) ammonia (0·880) in ethanol, 40°C for 30 min. Unextracted fabric. Liquor ratio 10 : 1.

† Bisulphite treatment—5% (w/v) sodium bisulphite in water, 40°C for 60 min. Extracted fabric. Liquor ratio 10 : 1.

EFFECT OF BISULPHITE TREATMENT ON THE FRICTIONAL PROPERTIES OF FIBRES

Samples of wool, unextracted or extracted with ethanol, were treated with 5% (w/v) sodium bisulphite in water at 40°C for 1 h. The frictional coefficients were determined, giving the results shown in Table XI. It is seen that there is no change in the difference between the frictional coefficients of the respective wools as measured by this method.

Discussion

The major points of interest which have emerged from these experiments are that it is possible to impart shrink resistance to wool by treatment with reducing agents, that preliminary ethanol extraction greatly enhances the effectiveness of such treatments when aqueous solutions are used, that the unshrinkability persists when many of the broken bonds are re-formed, and that the shrinkproofing effect is apparently unrelated to changes in frictional properties.

The preliminary investigations of the combination of disulphide reduction and peptide hydrolysis provide a foundation for interpreting the behaviour of ethanolic and aqueous solutions of thioglycollic acid and hydrazine or ammonia in terms of this same mechanism—combined disulphide and peptide breakdown. The need to have a relatively large excess of ammonia might be considered as supporting this idea, although the mild conditions which can give unshrinkability with ethanolic or aqueous solutions (25–40°C; 15–30 min) would not be expected to result in peptide hydrolysis. However, it is extremely unlikely that sodium bisulphite at pH 5·5 under the conditions used to give unshrinkability would have any effect on peptide bonds. Therefore, these results can only be accepted as confirmation of the contention of Speakman *et al.*[1, 2] that shrinkproofing agents

TABLE XI

Effect of Extraction and Bisulphite Treatment on Coefficients of Friction of Fibres

Treatment	Anti-scale coefficient	With-scale coefficient	Difference between coefficients
	Fibre and keratin rod, air-dry		
Unextracted—no treatment	0·24	0·17	0·07
Unextracted—bisulphite*	0·26	0·19	0·07
Extracted—no treatment	0·24	0·18	0·06
Extracted—bisulphite	0·28	0·20	0·08
	Fibre and keratin rod, wet		
Unextracted—no treatment	0·41	0·25	0·16
Unextracted—bisulphite	0·43	0·30	0·13
Extracted—no treatment	0·39	0·23	0·16
Extracted—bisulphite	0·55	0·40	0·15

* Bisulphite treatment—5% (w/v) sodium bisulphite in water at 40°C for 60 min. Liquor ratio 10 : 1.

which chemically attack the wool fibre are effective because they are capable of breaking disulphide bonds. This does not mean that any reagent capable of breaking disulphide bonds automatically renders wool shrinkproof— many results show that extensive attack on disulphides can occur without causing shrinkproofing [9, 20]. Furthermore, the results given in Table X show that the shrinkproofing effect may persist even if the disulphide bonds are re-formed.

It is generally believed that, for shrinkproofing to be achieved, the reaction must be confined to the surface of the fibre, resulting in modification of the frictional properties in such a way that the difference between the anti-scale and with-scale coefficients is greatly reduced. The ethanolic treatments described are unlikely to be surface reactions, and treatment with aqueous thioglycollate and bisulphite would certainly not be confined to the surface. Täuber [19], using staining tests to detect thiol groups, which would be formed by both thioglycollate and bisulphite, was unable to detect any preferential surface attack with either aqueous or ethanolic media. Furthermore, the results of Tables VIII and XI show that there is no correlation between shrink resistance and the difference between the two frictional coefficients. It might be argued that, although the difference between the coefficients, as measured by the present method, is unaffected by any of the treatments studied, the increase in both coefficients when measured wet on extracted fibres treated with bisulphite could account for the shrinkproofing effect in this instance. However, such an inference is invalidated by experiments [15] from which it is concluded that application of contaminants after the bisulphite treatment destroys the shrinkproofing effect without reducing either of the frictional coefficients or affecting the difference between them.

It appears therefore that, under certain undefined circumstances, breakdown of disulphide bonds in wool modifies some physical property of the fibre in such a way that fabrics made from the wool became unshrinkable. The requisite physical change may not be a modification of the surface or of the frictional properties, and it does not appear to be necessary for the disulphide bonds to remain broken for the effect to be maintained. These conclusions support other evidence [20] that, as proposed by Speakman *et al.*[1,2], attack on disulphide bonds is the only primary reaction necessary for chemical shrinkproofing, but the precise nature of the physical changes associated with this breakdown and which lead to unshrinkability has yet to be defined.

The effect of preliminary ethanol extraction, which enhances the effectiveness of shrinkproofing treatments with aqueous solutions of reducing agents, appears to be due to removal of a strongly adsorbed layer of wool wax. This is the subject of the following paper [15, 21].

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Fibre-surface Contaminants on Wool and Their Effect on Shrinkproofing

A. J. FARNWORTH

Normally processed wool fabric contains a small amount (about 0·5–1%) of wax (shown by Täuber to be strongly adsorbed components of wool wax) which is removed by extraction with alcohols but not by many other solvents. This wax contaminant affects resin adhesion, some shrinkproofing reactions, and dyeing rates. Ethanolic solutions of reducing agents and aqueous solutions of oxidising agents are able to shrinkproof unextracted fabric, despite the presence of residual wax. Their effectiveness is not due to removal of the natural contaminant during treatment.

Small amounts of lubricant-type surface contaminants, applied after mild chemical shrinkproofing treatments, restore the felting power of the treated wool. The effect appears to be due to modification of frictional properties by a lubricating action, although the restoration of the felting power occurs without alteration of the frictional coefficients measured by the capstan method. This raises doubts as to the relationship between such measurements and felting mechanism. More severe shrinkproofing treatments are generally unaffected by contaminants applied after treatment. Deliberately applied contaminants present before treatment can prevent the shrinkproofing reaction from occurring with both oxidising and reducing agents.

Introduction

Many methods have been developed for shrinkproofing wool by deposition of resins on the fibre surface. However, to obtain the maximum effect from such treatments, it is essential to have high resin–fibre adhesion, which can be achieved only by giving the wool some form of pretreatment before applying the resin [1,2]. The function of the pretreatment appears to be to clean or to roughen the fibre surface (or to do both), and one of the most effective pretreatments is extraction with ethanol [2].

In the preceding paper [3] it was shown that ethanol extraction also has a very marked effect on the degree of unshrinkability obtainable by treatment with aqueous solutions of reducing agents. It was decided, therefore, to investigate further the effect of ethanol extraction of wool, particularly in relation to its effect on shrinkproofing.

Although it is generally accepted that extraction of scoured wool with ethanol removes residual soap, many workers have shown the ethanol extract to be a complex mixture of soap, oxidised oil, higher alcohols, free and esterified sterols, and other components [4–6]. Other workers have reported that hot ethanol removes protein material [6,7] and modifies the surface of the fibre [1] even to the extent of causing the scales to become pitted [7]. Both ether and ethanol continue to extract material from wool even after several hundred hours [8].

Extraction with ether and ethanol greatly reduces the felting shrinkage of wool fabrics milled in acid solution, and application of lubricants to the extracted wool restores its ability to felt [9]. Speakman *et al.*[10] showed that the lubricating action of soap assists the shrinkage of wool fabrics during milling. Application of contaminants to extracted wool reduces the effectiveness of resins in preventing felting [1].

This paper describes experiments which show that extraction with ethanol removes fibre-surface contaminants, the presence of which inhibits satisfactory shrinkproofing, not only by resins but also by certain chemical methods. It is also shown that the application of small amounts of different contaminants to fabrics after shrinkproofing greatly modifies their shrinkage properties, and the presence of contaminants before treatment can prevent the shrinkproofing reaction from occurring.

Materials and Methods

The fabric used, and the methods adopted for measuring felting shrinkage and friction, were the same as those described in the preceding paper [3].

Solvent Extraction— Unless otherwise stated, fabric samples were extracted for 4 h in a Soxhlet apparatus, and then rinsed in running water.

Application of Contaminants— The various contaminants were applied from solvent solutions of known concentration. Unless otherwise stated, light petroleum (b.p. 60–80°C) was used as solvent. Fabric samples were immersed in the solution, then passed through a mangle adjusted to give a known retention of liquor, and finally air-dried. Samples were extracted periodically to check that the amount of contaminant applied was as calculated.

Treatment with Polyamide— This was carried out with *N*-methoxymethyl nylon, as described by Jackson [3].

Treatment with Casein— This was carried out as described by Jackson [12].

Shrinkproofing with Reducing Agents— Fabric samples were treated with one of the following solutions at 40°C for 1 h at a liquor ratio of 10 : 1, and then rinsed in running water—

(*a*) 0·25% (v/v) thioglycollic acid, 3% (v/v) ammonia (sp.gr. 0·880), in water;

(*b*) 1% (v/v) thioglycollic acid, 6% (v/v) ammonia (sp.gr. 0·880), in ethanol;

(*c*) 5% (w/v) sodium bisulphite in water.

Results

SHRINKPROOFING WITH RESINS

Effect of Extraction with Different Solvents

Fabric samples were extracted with different solvents prior to treatment with polyamide. Table I indicates the amount of material extracted and the area shrinkage after one and after five washes.

Although several solvents leave wool in a condition where resin treatment gives reasonable stability to moderate washing, only ethanol leads to stability to prolonged washing.

Table II shows how the effectiveness of the resin treatment was altered by varying the time for which fabrics were Soxhlet-extracted with ethanol. Stability to moderate washing is obtained by quite

TABLE I

Effect of Preliminary Extraction on Shrinkage after Treatment with Polyamide

Solvent	Extract (% on fabric weight)	Area shrinkage (%) after 1 wash	Area shrinkage (%) after 5 washes
Control	—	40	73
Unextracted	—	26	57
Ethanol	1·3	0	0
Benzene	0·7	5	43
Diethyl ether	0·4	18	47
Acetone	0·4	9	65
Chloroform	0·9	2	50

TABLE II

Effect of Duration of Ethanol Extraction on Effectiveness of Polyamide Treatment

Time of extraction (min)	Number of siphonings (approx.)	Total extract (% on fabric weight)	Area shrinkage (%) after 1 wash	Area shrinkage (%) after 5 washes
Control	—	—	40	74
Unextracted	—	—	23	65
15	1	1·0	3	32
30	3	1·2	2	17
60	6	1·3	1	6
120	12	1·4	0	4
240	25	1·4	0	5

short extraction (1 siphoning), but more prolonged extraction is required for stability to repeated washing.

Effect of Re-applying the Ethanol Extract to Extracted Fabric

After the fabrics had been extracted with ethanol, the solution of the extract was re-applied to clean fabrics previously extracted with ethanol. After being air-dried, the fabrics were treated with polyamide or casein, unextracted and extracted fabrics being included for comparison. Table III shows that the extract, when re-applied to extracted fabric, inhibits the effect of the resin treatments.

TABLE III

Effect on Resin Treatments of Re-applying Ethanol Extract to Extracted Fabric

Fabric	Treatment with polyamide Area shrinkage (%) after 1 wash	Treatment with polyamide Area shrinkage (%) after 5 washes	Treatment with casein Area shrinkage (%) after 1 wash	Treatment with casein Area shrinkage (%) after 5 washes
Control	40	73	40	71
Unextracted	26	57	18	59
Extracted	0	0	3	26
Extracted—extract reapplied	24	63	35	67

Jackson[1] observed that extraction in hot ethanol increased the rate of dyeing of wool with Naphthalene Scarlet 4RS (C.I. Acid Red 18), and inferred that this was due to modification of the fibre surface by hot ethanol. To test the validity of this inference, a fabric was extracted with ethanol and the extract then reapplied to another extracted sample. This "contaminated" fabric, the extracted fabric, and an unextracted fabric were dyed together in one bath with Naphthalene Scarlet 4RS at 50°C, in presence of Glauber's salt and sulphuric acid. There was a marked difference in the rates of dyeing of the three fabrics, the extracted one dyeing more rapidly than the unextracted which, in turn, dyed very much more rapidly than the contaminated fabric.

These results, together with those of Table III, suggested that surface cleansing is the predominant, if not the only, effect of ethanol extraction responsible for increasing the dyeing rate and enhancing the efficiency of resins as shrinkproofing agents.

Resin Treatment of Knitted Fabric Prepared from Solvent-degreased Dry-combed Wool

As ethanol extraction is usually considered to remove soap residues, and as Jackson[1] had shown that contamination of extracted fabric with soap inhibited shrinkproofing with resins, it appeared likely that the effects observed in the preceding section were due to removal of residual soap.

Knitted fabric was prepared from wool which had been solvent-degreased, rinsed with water to remove suint, and dry-combed. This fabric, which was therefore free from soap residues, was treated with polyamide and casein before and after extraction with various solvents. The results are given in Table IV.

TABLE IV

Effect of Extraction with Different Solvents on Shrinkage after Treatment with Polyamide or Casein

Solvent	Extract (% on weight of wool)	Area shrinkage (%) after treatment with polyamide	Area shrinkage (%) after treatment with casein
Control	—	40	40
Unextracted	—	14	37
Ethanol	2·5	1	7
Benzene	2·1	8	19
Diethyl ether	1·8	11	13
Acetone	2·2	11	15
Chloroform	2·7	5	19

Although these experiments were done on a different fabric, and the amount of extractable material is appreciably greater than that used in the experiments summarised in Table I, the results show that extraction in ethanol is more effective than extraction in other solvents in enhancing the efficiency of resin treatments, even when there is no possibility that soap residues play a part in the inhibition.

SHRINKPROOFING WITH SOLUTIONS OF REDUCING AGENTS

Effect of Extraction with Different Solvents

In the preceding paper[3] it was shown that ethanol extraction greatly enhances the shrinkproofing effect of aqueous solutions of reducing agents. Table V shows the effect of preliminary extraction with different solvents on treatment with aqueous solutions of thioglycollate or bisulphite.

Table VI shows that digestion (heating under reflux in a fixed volume) in boiling ethanol is also effective in promoting shrinkproofing, provided that there is a large excess of solvent.

TABLE V

Effect of Preliminary Extraction on Shrinkage after Treatment with Reducing Agents

Solvent	Extract (% on weight of fabric)	Area shrinkage (%) after treatment with thioglycollate	bisulphite
Control	—	40	40
Unextracted	—	27	36
Ethanol	1·3	5	4
Methanol	—	4	4
Benzene	0·7	11	12
Diethyl ether	0·4	—	22
Acetone	0·4	6	—
Chloroform	0·9	12	14

TABLE VI

Effect of Digestion in Ethanol on Shrinkage after Treatment with Sodium Bisulphite

Liquor ratio	Area shrinkage (%) after digestion at 40°C for 5 min	40°C for 30 min	78°C (boiling) for 5 min	78°C (boiling) for 30 min
Control	40	40	40	40
Unextracted	35	35	35	35
10 : 1	—	—	26	25
20 : 1	—	—	16	8
50 : 1	30	23	12	2
100 : 1	32	18	6	3

Digestion in other solvents is not as effective as digestion in ethanol, the order of decreasing effectiveness being ethanol > dioxan, chloroform, acetone > benzene, light petroleum, diethyl ether. Aqueous ethanol was effective so long as the concentration of water did not exceed 50% by volume.

Effect of Adding Contaminants to Extracted Wool before Shrinkproofing

At this stage of the investigation, it appeared likely that the reaction between wool and reducing agents responsible for the shrinkproofing effect was inhibited by the presence of some substance which could be removed by extraction with ethanol. To test this, different amounts of various contaminants were applied to ethanol-extracted fabric prior to treatment with aqueous bisulphite. The contaminants used (mineral oil, wool wax, oleic acid) were chosen as being typical of those most likely to be found on commercially processed wool. The results in Table VII show that very small quantities of contaminants have a marked inhibitory effect.

TABLE VIII

Effect of Contaminants Added to Extracted Fabric before Shrinkproofing with Ethanolic Thioglycollate

Contaminant	Area shrinkage (%)
None	1
0·1% mineral oil	2
0·1% wool wax	0
0·1% oleic acid	1
1% mineral oil	1
1% wool wax	5
1% oleic acid	3

Table VIII shows that contaminants added before treatment are without effect when shrinkproofing is obtained with ethanolic solutions of thioglycollate.

Effect of Adding Contaminants after Shrinkproofing Extracted Fabric with Reducing Agents

In the preceding work the fabrics were usually wetted out in water under vacuum, or in a 0·1% aqueous solution of Teepol and then thoroughly rinsed, before being shrinkproofed. To simplify and accelerate the procedure, it was decided to add a wetting agent to the bisulphite solution, which resulted in the observation that the presence of wetting agents (anionic, cationic, and non-ionic) appreciably reduced the degree of shrinkproofing. Although this did not invalidate the suggestion that contaminants in some way prevent the shrinkproofing reaction from occurring, it was decided to investigate the effect of adding the contaminants *after* the shrinkproofing treatment. In case immersion in Teepol for relaxation prior to washing had some complicating effect, a second set of fabrics was wetted out and relaxed in water only. The results obtained are shown in Table IX.

It is seen that application of wool wax to fabrics after treatment with reducing agents largely destroys the shrinkproofing effect. Fabrics relaxed in water were affected more than those relaxed in Teepol, probably because some of the wax was removed by the detergent solution.

Effect of Contaminating Fabrics after Shrinkproofing Treatments of Varying Severity

Investigation showed that fabrics shrinkproofed by McPhee's method, using potassium bromate in saturated sodium chloride[13], were unaffected by subsequent contamination with wool wax. In order to ascertain whether this was due to some

TABLE VII

Effect of Contaminants Added to Extracted Fabric before Treatment with Aqueous Sodium Bisulphite

Amount of contaminant (% on weight of wool)	Area shrinkage (%) when contaminant was soap applied from solution in water	mineral oil applied from carbon tetrachloride	wool wax applied from light petroleum	oleic acid applied from light petroleum
Control	40	40	40	40
Unextracted	34	34	34	34
Extracted— no contaminant	6	6	6	6
0·001	—	9	13	9
0·01	—	10	16	13
0·05	29	27	25	20
0·10	28	26	26	25
0·50	34	25	35	30
1·0	40	32	45	34

TABLE IX

Effect of Contaminant Added to Fabrics after Shrinkproofing with Reducing Agents

Shrinkproofing treatment	Area shrinkage (%) for series *relaxed in water*		*relaxed in Teepol*	
	not contaminated	contaminated with 1% wool wax	not contaminated	contaminated with 1% wool wax
Control	40	46	40	42
None	40	50	42	44
Aqueous bisulphite	6	35	6	25
Aqueous thioglycollate	4	36	8	25
Ethanolic thioglycollate	1	32	2	18

TABLE X

Effect of Contaminants on Fabrics Shrinkproofed at Two Levels of Severity

Contaminant	Area shrinkage (%) when contaminated after shrinkproofing with aqueous sodium bisulphite*		aqueous thioglycollate†		bromate–saturated sodium chloride [12]		permanganate–hypochlorite [18]		alcoholic alkali [19]	
	mild	severe	mild	severe	mild	severe	mild	severe	mild	severe
Control	40	40	40	40	40	40	40	40	40	40
None	10	2	4	2	20	1	20	3	20	9
0·1% mineral oil	9	3	5	3	17	2	16	4	23	10
0·1% wool wax	25	3	19	2	23	2	15	0	20	8
0·1% oleic acid	15	4	13	2	28	3	21	2	15	11
1% mineral oil	23	2	23	5	37	5	34	4	36	12
1% wool wax	37	9	50	17	35	0	21	2	28	17
1% oleic acid	21	1	14	2	18	4	25	2	17	10

* "Severe" treatment: 10% (w/v) sodium bisulphite; 55°C; 1 h.
† "Severe" treatment: 0·8% (v/v) thioglycollic acid; 7% (w/v) potassium carbonate; room temp.; 1 h [20].

fundamental difference between the effects of oxidising and reducing agents or to the relative severity of treatment with each method, fabrics were treated with different types of shrinkproofing reagents at two levels chosen as being mild and severe in each instance. The treatments used and the results obtained are shown in Table X.

All types of treatment show similar behaviour—a mild degree of shrinkproofing is nullified by contaminants, whereas a severe treatment usually gives a degree of shrinkproofing able to withstand the effect of contaminants. When reducing agents are used, small amounts of contaminant (e.g. 0·1% wool wax) can have a comparatively large effect.

From these results it seemed possible that the shrinkproofing effect was being upset by lubrication of the fibre surface. Speakman *et al.*[10] showed that soap can function as a lubricant in milling untreated fabrics. Therefore, since all washing tests in the present work were carried out in water (to avoid complications from the "scouring" effect of detergents), extracted fabrics, shrinkproofed with aqueous solutions of reducing agents, were washed in soap solutions.

Table XI shows the results obtained, together with those for similar fabrics washed in the usual way—i.e., relaxed in sodium bicarbonate–Teepol solution and washed in water. The shrink resistance of these fabrics is appreciably lowered when they are washed in soap solution.

TABLE XI

Effect of Washing Procedure on Shrink Resistance of Treated Fabrics

Shrinkproofing treatment	Area shrinkage (%) when washed* in water	0·05% soap solution	0·35% soap solution
None	21	17	28
Aqueous bisulphite	3	15	15
Aqueous thioglycollate	6	6	24

* Washing conditions— 20 min at 40°C.

Effect of Contaminating and then Extracting Fabrics before and after Shrinkproofing

Having established that contamination of fabrics either before or after treatment could destroy the shrinkproofing effect, we extended the experiments to ascertain the effect of removal of the deliberately added contaminant on shrinkproofing. There did not appear to be any reason why contamination, followed by removal of the contaminant *before* the shrinkproofing treatment, should have any inhibitory effect, and the results of Table XII show that good shrink resistance is obtained in these circumstances.

TABLE XII

Effect of Contamination followed by Removal of Contaminant before Shrinkproofing with Aqueous Bisulphite

Contaminant	Area shrinkage (%) when contaminated, then shrinkproofed	contaminated, extracted, then shrinkproofed
Control	40	40
0·1% mineral oil	23	8
0·1% wool wax	21	8
0·1% oleic acid	11	5
1% mineral oil	25	8
1% wool wax	28	7
1% oleic acid	33	8

Similarly, the shrinkproofing effect is restored when contaminants applied *after* shrinkproofing are removed by extraction (see Table XIII).

The more interesting point is whether application of the contaminant before the shrinkproofing treatment, and its removal *after* treatment, leads

TABLE XIII

Effect of Extraction of Fabrics Contaminated after Shrinkproofing with Aqueous Bisulphite

Contaminant	Area shrinkage (%) when fabric shrinkproofed, then contaminated	shrinkproofed, contaminated, then extracted
Control	40	40
0·1% mineral oil	19	8
0·1% wool wax	21	8
0·1% oleic acid	20	8
1% mineral oil	24	9
1% wool wax	29	9
1% oleic acid	27	9

to unshrinkability— i.e. whether the shrinkproofing reaction still occurs in presence of the contaminant, and its effect is revealed by extraction.

Table XIV shows results for fabrics contaminated before shrinkproofing with sodium bisulphite and then subsequently extracted. An original, unextracted fabric is included as representing one type of contamination. It is seen that the presence of contaminants prevents the shrinkproofing reaction from occurring. That this behaviour is not confined to treatments with reducing agents is shown in Table XV, where fabrics have been contaminated with wool wax before and after shrinkproofing with potassium permanganate in saturated sodium chloride solution [13]. Artificially applied contaminants again interfere with the shrinkproofing reaction when they are present before treatment, but have no effect when applied after treatment.

TABLE XIV

Effect of Contamination before Shrinkproofing with Sodium Bisulphite followed by Extraction after Shrinkproofing

Contaminant	Area shrinkage (%) before extraction	after extraction
Control	40	40
1% wool wax	40	38
1% oleic acid	44	39
1% mineral oil	35	28
Unextracted fabric	31	31

TABLE XV

Effect of Contaminant on Treatment with Potassium Permanganate

Treatment	Area shrinkage (%)
Control	40
Fabric extracted— shrinkproofed	3
Fabric extracted— shrinkproofed— contaminated with 1% wool wax	3
Fabric extracted— contaminated with 1% wool wax— shrinkproofed	21

Effect of Various Treatments on Amount of Extractable Material in Normal Fabric

It was previously shown [3] that preliminary ethanol extraction is not necessary for successful shrinkproofing with ammonium thioglycollate from ethanolic solutions. At first sight it would appear that the ethanol simply removes the contaminant from the wool during the reaction period. However, the results in Table VI show that ethanol alone under the conditions used for treatment with ethanolic thioglycollate does not predispose the wool to successful shrinkproofing with aqueous solutions of reducing agents.

To investigate this point further, unextracted fabrics were treated with ethanol, ethanolic ammonia, or ethanolic thioglycollate under the conditions used for shrinkproofing with the last-named reagent. The amount of material extractable by light petroleum and ethanol, respectively, was then determined on the treated fabrics. Similar extraction experiments were carried out on fabrics treated with potassium bromate or potassium permanganate in saturated sodium chloride solutions, since these treatments, under the conditions defined by McPhee [13], shrinkproof wool without preliminary extraction with ethanol.

The results in Table XVI show that ethanol alone has only a slight effect on the extractable matter, ethanol plus ammonia or thioglycollate very markedly reduces the amount of ethanol-extractable material, and the oxidising agents, particularly potassium bromate, increase the amount of material extractable by light petroleum at the expense of the ethanol-extractable material, the total amount being unaltered.

TABLE XVI

Amount of Extractable Material remaining on Unextracted Fabric after Various Treatments

Treatment*	Light-petroleum extract (% on dry weight of wool)	Ethanol extract
None	0·1	1·4
Water	0·1	1·3
Ethanol	0·0	1·1
Ethanol + 6% (v/v) ammonia	0·0	0·5
Ethanol + 1% (v/v) thioglycollic acid, 6% (v/v) ammonia	0·0	0·3
Potassium bromate in saturated sodium chloride solution [13]	0·7	0·9
Potassium permanganate in saturated sodium chloride solution [13]	0·3	1·0

* All treatments except with bromate and permanganate carried out at 40°C for 30 min at liquor ratio 10 : 1.

Effect of Contaminants on Frictional Properties of Fibres

Samples of wool, extracted with light petroleum and ethanol, were contaminated with wool wax, oleic acid, or mineral oil both before and after treatment with aqueous sodium bisulphite. The coefficients of friction were determined, giving the results shown in Table XVII. Addition of contaminants to either extracted or extracted and shrinkproofed fibres has no significant effect on the difference between the coefficients.

Discussion

This investigation has shown that the presence of fibre-surface contaminants on wool can seriously interfere with shrinkproofing, not only by resins but also by some chemical treatments. The interference can arise from residual material retained by wool after normal processing, or from contaminants applied to wool previously purified by ethanol extraction.

TABLE XVII

Effect of Contaminants on Frictional Properties of Extracted and Bisulphite-treated Fibres

Treatment	Coefficient of friction					
	In air			*In borate–Teepol solution*		
	Anti-scale	With-scale	Difference	Anti-scale	With-scale	Difference
None	0·24	0·17	0·07	0·41	0·25	0·16
Extracted	0·24	0·18	0·06	0·39	0·23	0·16
Extracted + 0·1% wool wax	0·25	0·18	0·07	0·43	0·28	0·15
Extracted + 0·1% oleic acid	0·28	0·19	0·09	0·42	0·23	0·19
Extracted + 0·1% mineral oil	0·29	0·21	0·08	0·35	0·23	0·12
Extracted + 1% wool wax	0·23	0·17	0·06	0·35	0·23	0·12
Extracted + 1% oleic acid	0·26	0·19	0·07	0·44	0·24	0·20
Extracted + 1% mineral oil	0·26	0·19	0·07	0·42	0·24	0·18
Extracted— bisulphite	0·28	0·20	0·08	0·55	0·40	0·15
Extracted— bisulphite— 0·1% wool wax	0·28	0·23	0·05	0·48	0·36	0·12
Extracted— bisulphite— 0·1% oleic acid	0·25	0·21	0·04	0·54	0·40	0·14
Extracted— bisulphite— 0·1% mineral oil	0·25	0·21	0·04	0·52	0·37	0·15
Extracted— bisulphite— 1% wool wax	0·26	0·21	0·05	0·50	0·38	0·12
Extracted— bisulphite— 1% oleic acid	0·27	0·23	0·04	0·53	0·38	0·15
Extracted— bisulphite— 1% mineral oil	0·27	0·23	0·04	0·52	0·40	0·12

Täuber[14] has investigated the composition of the ethanol extract of wool after cleansing with light petroleum and water, and has found it to consist of strongly adsorbed wool-wax components. There appears to be little doubt, therefore, that the natural contaminating material is a component of wool wax tenaciously held by the fibre, and removable only by extraction with certain solvents. Further evidence for this is given by the results of Table IV, where fabric, prepared in such a way that it could not contain any contaminants other than those naturally occurring, could be shrinkproofed with resins only after extraction with ethanol.

Of several solvents investigated, ethanol is the most effective; resin adhesion sufficiently strong to withstand prolonged washing, or effective shrinkproofing with aqueous solutions of reducing agents, is possible only after extraction with ethanol. This raises the possibility that the effect is due to surface modification of the fibre, since various workers have claimed that ethanol extraction can have marked effects on the surface of the wool fibre[1,6,7]. Whether or not extraction under the conditions used brings about such changes, they do not appear to play any part in the effect considered here, as redeposition of the extract destroys the advantage gained by extraction (Table III). Furthermore, the increased rate of dyeing given by ethanol extraction, and claimed to be due to surface modification, is completely lost by redeposition of the extract on previously cleaned wool (see p. 490). It appears very probable, therefore, that the small amount (ca. 0·5–1%) of strongly adsorbed wool-wax components retained by normally processed wool[14] interferes with resin adhesion, shrinkproofing with aqueous solutions of certain reagents, rate of dyeing, and possibly other properties of wool. The wax is removed by alcohols but not by many other solvents. These observations form an interesting contrast to the effect of ethanol extraction before treatment with sulphuryl chloride in carbon tetrachloride. In this instance, extraction renders the treatment *ineffective* by removing the peroxides which catalyse the reaction of the wool with sulphuryl chloride[15].

When the general effect of contaminants, both natural and applied, is considered, there appear to be two entirely different mechanisms by which they can nullify the shrinkproofing effect produced by chemical treatments. Firstly, contamination *after* treatment destroys the shrinkproofing effect when the treatment has been mild, but has no effect for more severe treatments (Tables IX and X). That this is purely a mechanical effect, and not one of chemical modification by the contaminant, is shown by the fact that, when shrinkproofed fabric has been contaminated, the shrinkproofing effect which is then lost can be restored by removal of the contaminant by extraction (Table XIII). Treatments with reducing agents are, in general, more sensitive to contamination than are those with oxidising agents, as little as 0·1% of wool wax being sufficient to nullify the effects of treatments with mild aqueous bisulphite or thioglycollate. The sensitivity of the shrinkproofing effect obtained with these reagents is also demonstrated by the fact that the shrink resistance of the fabric washed in water is appreciably reduced when soap is present in the wash liquor (Table XI). McPhee[16] has further studied this effect and has observed a similar behaviour with some other shrinkproofing treatments.

The contaminants used are generally regarded as being lubricants for metal–metal interfaces, and although it is by no means certain that they are also lubricants for wool against wool, it seems reasonable to assume that their effect when added after shrinkproofing is one of lubrication. The general mechanism for shrinkproofing by chemical treatments has been accepted as a surface modification with consequent change in frictional properties. With mild treatments, it might be expected that the degree of surface modification would be slight, and the effect on the frictional properties can then be masked or nullified by adding a lubricant, thus restoring the felting properties. When the shrinkproofing treatment is more severe, however, the modification may be too great to be affected by lubrication, and the non-felting properties persist. The difference in

the effect of lubricants after mild and severe treatments is more in agreement with the directional frictional effect being mechanical in origin rather than molecular as proposed by Martin [17], who suggested that the frictional properties of wool are due to the molecular orientation at the scale edges. If this were the case, it might be expected that the layer of lubricant would mask these molecular forces, irrespective of the degree of chemical attack during shrinkproofing.

If the contaminants used in this work do, in fact, restore the felting power by overcoming frictional forces through lubrication, the results of measurements of coefficients of friction (Table XVII) are of interest and importance, since the contamination treatments which destroy the shrink resistance are completely without effect on the coefficients measured by the present method. This implies that felting of wool involves frictional forces which are unrelated to those measured by this method.

The second mechanism by which contaminants can nullify the shrinkproofing effect of chemical treatments arises when the contaminant is present *before* shrinkproofing. The results of Tables V, VII, and XV show that both natural and applied contaminants can interfere with a shrinkproofing treatment, even when present in only small quantities, and that treatments with either reducing or oxidising reagents can be seriously impaired. Initially it was thought that this effect was identical with that discussed above— the contaminant withstood the shrinkproofing treatment and functioned as a lubricant as before. However, the results of Table XIV show that this is not so, because removal of the contaminant after shrinkproofing does not give an unshrinkable fabric. It appears, therefore, that the contaminant prevents the shrinkproofing reaction from occurring, and this same behaviour is shown in Table XV, where wool wax applied to fabric after treatment with permanganate in saturated sodium chloride is without effect, but wax applied before treatment seriously impairs the shrinkproofing effect.

Ethanolic treatments with reducing agents are not affected by the presence of either natural or applied contaminants before shrinkproofing (Table VIII and preceding paper [3]) and, furthermore, natural contaminants do not interfere with aqueous treatments with oxidising agents [13], in contrast to treatments with reducing agents. It was thought that the contaminant might be removed during these treatments, thus facilitating the normal reaction. Table XVI shows that the contaminant is not dissolved by ethanol under the reaction conditions used, but is apparently largely desorbed, dissolved, or otherwise removed by ethanolic ammonia or ethanolic thioglycollate. Despite this, it is highly improbable that the inability of contaminants to interfere with the ethanolic treatments can be explained in terms of their removal during treatment, since although the amount of extractable material retained by the wool is only 0·3–0·5% (Table XVI), it is still appreciably greater than the small quantities which have been found to interfere (Tables II, V, VII). Furthermore, it has been found that pretreatment of fabric with ethanolic ammonia does not lead to shrinkproofing when the fabric is subsequently treated with aqueous sodium bisulphite. Aqueous treatments with oxidising agents, whilst having some effect on the relative proportions of material extractable in light petroleum and in ethanol, respectively, do not reduce the total amount of contaminant.

* * *

Grateful acknowledgment is made to Dr. M. Lipson and Dr. J. R. McPhee for helpful discussion, and to Mrs. Valeria Heyburn and Messrs. B. D. Harding and K. I. M. Kelaart for carrying out most of the experimental work described in this and the preceding paper.

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Crease Recovery

Since I drew attention to some anomalous results from tests of dry and wet recovery from creasing [1], various correspondents in Europe and America have signified their agreement; the suggestion has also been made that a fuller discussion might be opportune.

It seems to me that insufficient consideration has been given to the importance of hydrogen bonding in the variously treated products, particularly during washing and drying without ironing. Their fundamental impact on crease recovery is shown in the behaviour of untreated cotton, which exhibits good crease recovery when completely dry but loses this property as the cotton absorbs moisture again; this is due to the making and breaking of hydrogen bonds.

In the conventional treatment with amino-aldehydes at temperatures around 150°C, the chain-molecules in the amorphous regions of cellulose become entangled by "resin bonds" supplemented by hydrogen bonds [2]. When a fabric is creased or distorted, some of the force of deformation is thrown on these entanglements or linkages, and so establishes a recovery mechanism which may be likened to that of the self-closing door. The importance of the hydrogen bond in conventional crease-resisting goods is shown by the fall in recovery as their moisture content increases [3]; in atmospheres of 90% r.h. it is less than 90% of that in atmospheres of 65% r.h. The question naturally arises as to why these products do not exhibit much worse crease recovery when wet; indeed, there are many examples where the crease recovery in the wet state is slightly better than that in atmospheres of 90–95% r.h. This is probably due to a new factor, namely, the swelling of the treated material in water; although this is less than that of the untreated cotton (water imbibition of 60%) it is still appreciable (water imbibition of 40%). Distortion by external forces in the wet state is resisted by the internal swelling pressure; when the wet fibre is deformed, the original unstrained state is restored when the stress is released, as may be seen with the lively and resilient canvas hose-pipe filled with water.

Hence there is a complicated interplay of forces in the "resin-treated" crease-resisting goods when wet; the resin bonds persist, many of the hydrogen bonds are broken, but the crease recovery in the wet state is assisted by the water of imbibition which compensates, to some extent, for the breakdown of hydrogen bonds. In general, the "dry-recovery" cottons all possess an appreciable amount of wet recovery.

Since 1954 there has been increasing interest in the commercial promotion of "wash-and-wear" fabrics, and this has caused attention to be concentrated on the property of wet recovery; the autumn of 1958 saw the development of cotton goods which possessed wet recovery but little or no dry recovery. The "wet-recovery" processes depend fundamentally on setting the fibre in a swollen state, and the extent of swelling at the time of setting determines the degree of wet recovery. In the swollen state, those hydrogen bonds present in the dry state are absent, so that the chain-molecules are able to move apart and any linkages which are placed between them must be much nearer to the crystallites than is the case with the amino-aldehyde processes. Furthermore, the wet-recovery processes operate at room temperatures, so that for these two reasons the final product is lacking in hydrogen bonds, compared with the conventional product, and the distribution of chemical bonds is quite different. Fundamentally, the wet-recovery processes may be regarded as disentanglement processes; the wet-recovery product has a high swelling value, the water imbibition being 20–25% greater than that of the untreated cotton.

The explanation of wet recovery only, as distinct from dry recovery accompanied by wet recovery, was put forward in 1959 [4] and has since received support from other workers in this field [5]. It does not seem to be appreciated, however, that good wet recovery can result from two such different systems, namely, entanglement with diminished swelling on the one hand, and disentanglement with increased swelling on the other. When the same test is used on such different products, and also for dry recovery, it is not surprising that anomalous results are obtained.

Suggestions that a high degree of wet recovery is the most important factor in predicting smooth-drying performance [6] have not been confirmed in actual practice and, indeed, there is a growing body of opinion which considers that tests of angular recovery, wet or dry, correlate poorly with visual assessment of a washed sample [7]. More reliance is being placed on some form of integrated test, objective or subjective, which not only includes the number of creases produced by mechanical deformation, but also the wrinkles due to the release of internal stress when the fabric is wetted and then subjected to free drying.

In our present state of knowledge, it would be unwise to insist that either wet recovery or dry recovery, or both, will determine good smooth-drying behaviour; indeed, there is a very good case for assuming that the most important single factor is recovery *during* drying. This can be shown by wetting a sample of crease-resisting fabric, squeezing it in the hand, and then carefully placing it on a flat surface, where it is allowed to dry without disturbance. Many of the imparted creases will persist, owing to the formation of hydrogen bonds as the fabric dries; their power may be seen by manipulation of the dried fabric, concertina-wise, when the hydrogen-bonded creases neutralise a substantial amount of the crease-recovery effect imparted by the amino-aldehyde bonds or entanglements. These hydrogen-bonded creases will disappear when the fabric is passed through another

moisture cycle, but will come and go according to the conditions of drying, which can operate with or against wet recovery and dry recovery. Many of the integrated tests of wash-and-wear performance reflect these conditions of drying rather than the power of crease recovery.

At the present time, there are three main types of home laundering to which self-smoothing fabrics are subjected—

(*a*) hand-wash, squeeze, and drip-dry
(*b*) machine-wash, spin, and line-dry
(*c*) machine-wash, spin, and tumble-dry.

The drip-dry technique is at its best with fabrics possessing either good wet recovery or good wet and dry recovery; in addition to wet and/or dry recovery imparted by chemical finishing, the swollen cloth is heavy with water, so that, when it is freely suspended vertically, its own weight exerts an additional self-smoothing effect which is influential in the plastic state, and the hydrogen bonds re-form on drying in the smooth fabric. Spin-dryers (centrifuges) are more drastic in their effect and can offset some of the benefits of the chemical finish, particularly when the cloth is left in the damp state and full of creases; the tumble-dryer will remove these where the amino-aldehyde finish has been used, for the dry-recovery effect is very powerful with the warm, dry fabric, but the product with only wet recovery is badly creased in the tumble-dryer. (The making of hydrogen bonds is, of course, affected by temperature as well as by humidity.)

The modern spin-dryer is designed to promote rapid drying by removing as much water as possible; in certain circumstances this leaves the fabric full of imparted creases and without a weight of water sufficient to pull them out during free drying. It is these "dried-in" creases that are mainly responsible for crease-retention after laundering; they are set in a semi-permanent state by hydrogen bonding in the amorphous regions of cellulose. These bonds, whose existence is so often neglected or even rejected by textile chemists, help to make or mar the appearance of the smooth-drying fabric.

It is clear that crease recovery in the region around 100% r.h. is very important, but it would be difficult to measure accurately because of the onset of capillary condensation with the complication of crease recovery due to swelling; the region of 90–95% r.h. would be free from these objections, and as it is here that all cellulose fibres, whether treated or untreated, exhibit minimum recovery, a good recovery in this region would be accompanied by a better recovery in either the dry or wet state. High-humidity recovery appears to be worthy of attention as an index of wash-and-wear performance.

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Notes

Publication of Symposium Papers

The papers presented at the Bristol Symposium on "Science and Craft in both Textile and Non-textile Coloration" will be published in the December issue of the *Journal*, copies of which will be available at a special price to non-subscribers.

Liaison Officers

A suggestion was made at the last meeting of Section Chairmen and Honorary Secretaries that each Section should appoint a Liaison Officer to assist Publications Committee to obtain manuscripts of suitable lectures with a view to their subsequent publication in the *Journal*. The Manchester Section has appointed Mr. H. A. Turner to act in this capacity.

Perkin Centenary Trust

Applications for awards for the academic year 1962–63 should be made before 1st May 1962 on forms available from the Secretary, The Perkin Centenary Trust, c/o The Chemical Society, Burlington House, London W.1.

Perkin Centenary Fellowship—For one or two years' higher study of any approved subject. Candidates must have had experience in the manufacture or application of colouring matters, or their intended field of study must have a direct bearing on these subjects. The value of the Fellowship is £750 p.a., with an additional grant of up to £100 p.a. towards certain designated expenses. It may be tenable, from October 1962, at any university, technical college or other institution approved by the Trustees.

Perkin Centenary Scholarships (2)—For two years, renewable at the discretion of the Trustees for one further year, to enable candidates employed in the manufacture or application of colouring matters to study at a university or technical college. Each award will have a value of £350 per annum. This may be increased to £400 per annum if the candidate appointed is required to live away from home.

Olney Medal

The 1961 Olney Medal of the American Association of Textile Chemists and Colorists was presented to Mr. Fred Fortess at the AATCC National Convention in Buffalo, N.Y. on 28th September. Mr. Fortess, who is manager of the Dyeing and Finishing Laboratories, Celanese Fibers Co., Charlotte, N.C., has published original work on the

mechanism of dyeing of hydrophobic fibres with disperse dyes and on the mechanism of silicone finishing and its effect on textile properties. He has also worked on fermentation and wood-pulping by-product utilisation and on the development of yarn and fibre lubricants and antistatic agents. Mr. Fortess is Vice-Chairman of AATCC's Technical Committee on Research and a member of the Association's Executive Committee on Research.

British Patent Applications (Amended Rules)

At present a patent application must be put in order for acceptance within three years and six months after the date of filing the complete specification. This will continue to apply to applications filed before 1st January 1962. In the case of an application filed after that date, the period will be three years.

On Complete Specifications filed on or after 1st October 1961, the Stamp Fee will be £10, instead of £4.

Out of the Rut

In an essay that is in effect a review of the book *Light, Colour and Vision* by Yves le Grand (Pp. xiii + 512. London: Chapman and Hall. 1957. 63s.), H. C. F. Usherwood draws attention to the stagnant and conservative character of most university syllabuses. Too much time is devoted to subsequently useless and purely historical material; the thermionic valve, for instance, is just beginning to find a place in courses and textbooks—at a time when it is yielding place to semi-conductors. Despite the immense importance of colour in daily life in matters ranging from art, advertising, and printing, to textiles, paints, and colour television, and in science and technology in the growth of colorimetry, the study of light is still confined almost exclusively to geometric optics and the determination of the speed of light. Few science graduates would be in a position to discuss the Munsell and Ostwald systems. The excuse that colour is a subjective sensation is not valid, and colorimetry at least would be unaffected by the disappearance of colour vision from the human race. The work under review is described as a "timeless" textbook in a branch of pure physics that should receive very much more attention than at present (*Chem. and Ind.*, No. 28 (15 July 1961) 1066). J.W.D.

Modern Approach to Laboratory Planning

Ideally a laboratory building should be regarded as a cladding around the optimum layout of benches and services, but this is seldom possible. The planning of both new laboratory buildings and conversions of existing premises is discussed. It is recommended that a floor-space of 130–150 ft² per worker is appropriate in laboratories devoted to routine work, whereas 230 ft² is more appropriate in research establishments; room taken up by ancillary services and staff and by communicating corridors is not included in these figures. Planning of layout is treated in some detail; the relative merits of open-planning and division into bays or separate rooms are considered, together with the siting of ancillaries, e.g. wash-ups, balance benches, and fume cupboards. Fixed benches are suitable for routine laboratories, but there is a strong case for deployable unit furniture in research laboratories, despite the higher cost. The use of cheap materials for bench tops, drains, and the like is a false economy, and careful attention to the planning of services is of great importance. The design and choice of materials for benches is considered at length. Fume cupboards present a potential health hazard and their design in relation to efficiency and easy operability is treated fully, with four diagrams and two photographs. (P. J. Skinner, *Chem. and Ind.*, No. 29 (22 July 1961) 1099–1105.) J.W.D.

U.S. Production of Dyes and Pigments in 1960

The U.S. Tariff Commission reports that production in the U.S.A. of many important dyes was lower in 1960 than in 1959. Thus, production of C.I. Vat Blue 1 was down by 14% (7·7 million lb in 1960 compared with 8·9 million lb in 1959), C.I. Direct Black 38 by 5% (5·6, 5·9), C.I. Mordant Black 17 and C.I. Vat Red 1 each by 53%, C.I. Mordant Black 11 by 45%, C.I. Vat Blue 6 by 32%, C.I. Basic Violet 1 and C.I. Disperse Black 9 each by 30%, C.I. Direct Yellow 6 by 25%, and C.I. Direct Black 80 by 24%. On the other hand, production of some dyes was higher, e.g. C.I. Vat Black 25 by 39% (2·5 million lb compared with 1·8 million lb), C.I. Vat Black 27 by 59%, C.I. Vat Green 3 and 8 by 30% and 46%, respectively, and C.I. Vat Brown 3 by 24%.

Production of full-strength and extended toners and lakes was 40·3 million lb (42·7 and 35·4 million lb in 1959 and 1958, respectively), sales being 32·7 million lb, and of value $64·3 million. Full-strength toners in 1960 amounted to 30·5 million lb, among the chief being C.I. Pigment Red 49 (Ba lake) (2·8 million lb), C.I. Pigment Yellow 12 (2·5), C.I. Pigment Blue 15 (α-form) (2·3), C.I. Pigment Green 7 (2·2), and C.I. Pigment Red 3 (2·1). C.O.C.

Eighth Canadian Textile Seminar

The Eighth Seminar will be held at Queen's University, Kingston, Ont., on 5th–7th September 1962. Seminar sessions will be conducted on the morning and afternoon of the Thursday and Friday. The Seminar is divided into five sections: Conversion of Fibres into Yarns; Conversion of Fibres and Yarns into Fabrics; Dyeing and Finishing; Mill Supervision; and Knitted Fabrics.

A departure from past procedure will be the Federation luncheon, scheduled for September 6, at which the Keynote Address will be delivered and the Federation's Meritorious Service Award will be presented.

Meetings of Council and Committees

September

Council— 6th
Finance and General Purposes— 12th
Light Fastness Subcommittee— 14th
Colour Index Editorial Board— 25th
Publications— 26th
Data on Identification of Dyes on Textile Fibres— 29th

OBITUARY NOTICES

Professor W. T. Astbury, F.R.S.

William Thomas Astbury was born at Longton, Stoke-on-Trent, in 1898, and obtained a Science Scholarship to Jesus College, Cambridge, from Longton High School. He was awarded a First in both Part I (Chemistry, Physics, and Mineralogy) and Part II (Physics) of the Natural Sciences Tripos, and on graduation in 1921 took up an appointment as Demonstrator in Physics at University College, London. Then followed a period as Assistant to Sir William Bragg at the Davy–Faraday Laboratory of the Royal Institution from 1923 to 1928, and it was his specialised training there in X-ray crystallography that he brought to the service of wool when he joined the staff of the Department of Textile Industries of the University of Leeds as the first Lecturer in Textile Physics. Among the many papers embodying the results of his early years' work at Leeds were two in the Philosophical Transactions of the Royal Society which laid the foundations of far-reaching developments in fibre physics and textile technology. The progress of this work on wool was the subject also of several lectures to the West Riding Section of the Society, and it is interesting that the first recognition of the profound theoretical and practical importance of Astbury's discoveries should have been the award of the Worshipful Company of Dyers Gold Research Medal for the paper which he wrote for the Jubilee Number of the *Journal* of the Society in 1934.

Although Astbury never ceased to be fascinated by the wool fibre and its complexity, it was not to be expected that a single protein could long contain his genius. From 1934 onwards he was encouraged by generous grants from the Rockefeller Foundation to extend his studies of keratin to other proteins, and in this way he came to classify the fibrous proteins into two configurational families—the k-m-e-f group and the collagen group; to recognise the similarity between the keratins of birds and reptiles, which distinguishes them from mammals; to demonstrate the common configurational plan embodying the elastic and contractile properties of hair, epidermis, muscle, and bacterial flagella; to interpret protein denaturation and the relation between fibrous proteins and "globular" proteins; and to initiate studies of the structure of nucleic acids and nucleo-proteins. He founded the subject of "molecular biology" and, in the words of Professor C. H. Waddington, "opened the doors through which so many people are now passing". During these later years Astbury's triumphal progress was marked by a series of distinctions: the Actonian Prize of the Royal Institution in 1935, election to the Fellowship of the Royal Society in 1940, appointment to a personal Chair of Biomolecular Structure at Leeds in 1945, election to the Royal Society of Sciences, Uppsala, in 1946, and election to the New York Academy of Sciences in 1950. Early this year he was chosen as the fourth recipient of the T. Duckett Jones Memorial Award in the United States for "pioneer and continuing work in the fields of X-ray diffraction of fibrous structures". Astbury's fear was that a recurrence of the illness which overtook him in Australia in 1955 might lead to his death before the presentation could be made, and this fear, alas, proved to be justified, for he died on June 4th at the age of sixty-three.

But such an account of achievements and distinctions does nothing to recall the vivid personality we have known. Each of us has special memories of the gusto that he brought to scientific research, which once led a colleague to say that "to be with Astbury is like a perpetual Christmas morning"; each of us could give examples of the wit which enlivened all his activities, such as the remark about a student to whom he had given the unprecedented mark of 98% in a Textile Physics examination: that "he ought perhaps to have been given 100%, but that would have meant that he is as good as I am, which is ridiculous"; and, finally, many will remember his love of music, especially that of Mozart and Beethoven, which was the solace of the years when he knew that his life was threatened. We rejoice in a life that has been so rich in achievement and friendship and, great as is our loss, we may still say:

Nothing is here for tears, nothing to wail
Or knock the breast; . . . . . .
. . . . . nothing but well and fair.

J. B. Speakman

G. E. Styan

George Eric Styan, Chairman of the West Riding Section in 1957–8, and Honorary Secretary of the West Riding Section from 1951 to 1956, died in Bradford on 5th July 1961 at the early age of 50. Born on 4th April 1911, in Harrogate, Eric Styan was educated at Bradford Technical College, with which institute he was associated continuously as student, research worker, and lecturer for 34 years. In 1927 he joined the firm of John Smith & Sons Ltd., Fieldhead Mills, Bradford, as Assistant Chemist, and rose to the position of Chemist–Dyer, in charge of the laboratories and chemical control of combing plant operations, grease-processing plant, and the slubbing dyehouse. He stayed with this firm until 1948, when he joined the staff of what was then the Department of Chemistry and Dyeing of Bradford Technical College, as Senior Assistant Lecturer in Dyeing and Dyestuffs Chemistry, and was promoted to Senior Lecturer in 1951.

On the designation of the College as a College of Advanced Technology, Styan was promoted to Principal Lecturer in Dyeing and Dyestuff Technology, and assumed detailed control of the Dyeing Section of the Department of Chemical Technology of Bradford Institute of Technology.

Throughout the whole period of his association with the College at Bradford, Styan had steadily

pursued an ambition to equip himself to the fullest extent with the scientific and academic basis which he considered essential for the fullest development of his own technology of dyeing. Graduating as an external student of London University in 1942 with the Special B.Sc. in Chemistry, with honours, Styan proceeded to carry out research on a part-time basis, in collaboration with Dr. (now Professor) R. M. Barrer, on the subject of gaseous adsorption on zeolites. After 1947 Styan undertook research in collaboration with his colleague, Dr. W. R. Moore, and publications appeared on each of these researches.

In 1954, Styan was elected a Fellow of the Society, and in 1960 a Fellow of the Royal Institute of Chemistry.

During the period from 1947 until his death, Styan was a most active worker for the Society, initially as Honorary Secretary of the West Riding Section, where his pleasant and quiet efficiency did much to enhance the reputation of the Section. Later, as its Chairman, he added a dignity to the proceedings of the Section, without destroying the spirit of goodwill which permeated everything with which he dealt.

He was an active member of the Publications Committee from 1952 until his death and of the Committee for Data on Identification of Dyes on Textile Fibres from 1957, and did much good work outside of committee for both. Styan was also a most willing contributor to the Society's Summer Schools, and he will be remembered by all who attended the School at Nottingham in particular, for the excellence of the practical work provided, and for the mature and efficient manner in which he gave guidance in the laboratories. Styan was responsible for devising, organising, and controlling the work.

Styan's death, after such a brief illness, came as a great blow to all who were associated with him, and his passing is a great loss to the dyeing community at large. More particularly, he will be missed by those closely associated with him, both in the affairs of the Society and in the educational field to which he had devoted so much of his time and energy. He was a most courteous, pleasant, and conscientious colleague, always on the most friendly terms with everyone, and always willing to give of his advice and service.

His only hobby was gardening, and his enthusiasm and skill in this also gave added pleasure to his family and his friends. He is survived by his wife and two daughters.

R. L. ELLIOTT

N. Ellse

Norman Ellse, of Burley-in-Wharfedale, who died very suddenly on July 6th, was associated with Sandoz Products Ltd, of Horsforth, for 47 years. He began as office boy in 1914, when premises were occupied in Arundel Street, Bradford, and eventually became Dyestuff Sales Manager, a position which he held until he retired owing to ill health at the end of June this year. He frequently attended meetings of the West Riding Section of the Society.

Ellse was a member of the Acacia Lodge of Freemasons for 30 years, becoming Worshipful Master in 1949 and 10 years later receiving Provincial Honours. He was a member of the Northcliffe Golf Club for 30 years, occupying during that time the offices of Captain and President. He had been actively engaged in the Rotary Organisation since 1953 and was specially interested in the broadcasting of sporting events to local hospitals.

Ellse leaves a widow, and a son and a daughter, both of whom are married.

W. Meitner

Walter Meitner, who died on July 6th at the age of 70, was a member of the Society for many years. He came to England in 1938, from Austria, and was at one time engaged in research work on dyeing and printing problems at I.C.I. Dyestuffs Division, Blackley, and later at the B.R.R.A. He studied particularly the mechanism of fixation of dyes on rayon (J.S.D.C., **61**, 33 (1945)). His work was mentioned by G. S. J. White in the Fourth George Douglas lecture as one of the five basic contributions to the better understanding of the mechanism of dyeing. He collaborated with H. B. Hampson in the use of radioactive isotopes to investigate practical problems in roller printing, and an account of this work was published in the *Journal* (**69**, 283 (1953)).

For the last year or so Meitner had served as an Abstractor, dealing particularly with foreign-language publications.

New Books and Publications

Azo and Diazo Chemistry: Aliphatic and Aromatic Compounds

By H. Zollinger. Pp. 444, with 25 tables and 13 fig. in the text. New York: Interscience Publishers, Inc. 1961. Price, 124s. 0d.

This well-produced book will fill a long-felt need for a clear and concise account of the whole field of diazo chemistry, not only by the dye technologist but also by the so-called pure organic chemist. The most recent publications (1947) have been concerned mainly with aromatic diazo compounds, and none dealing with diazo- and azo-alkanes has so far been forthcoming. The author states in the preface that the time now seems ripe for an examination of these classes of compound from the common viewpoint of physical organic chemistry, and he certainly succeeds in proving this suggestion. Kinetics of diazotisation, isomerism and acid–base equilibria in diazo compounds, conclusions drawn from kinetic isotope effects in azo coupling, and the

stereochemistry and stability constants of metal complexes containing the azo linkage may be quoted in support of this viewpoint.

This is not a monograph on dye chemistry, although the chapters on aromatic diazo and azo compounds have been translated with additions from the author's book *Die Chemie der Azofarbstoffe* (J.S.D.C., **75**, 156 (1959)). The tendency in translation to retain the same grammatical construction has been successfully overcome. The text is easily read and amply illustrated with modern structural formulae. The amount of effort put into the preparation of this work may be judged from the number of references to original research papers (2389), whereas references to patent literature have been kept to a minimum (103).

Division of the contents falls readily into two main parts, the first seven chapters being devoted to aliphatic and aromatic diazo derivatives and the last six to the preparation and properties of azo compounds, with an intermediate chapter on the diazoamino derivatives. The preparation of diazonium salts and diazoalkanes, and modern concepts of the mechanism of diazotisation are lucidly dealt with in the first two chapters, while Chapters 3 and 4 deal with acid–base equilibria and isomerism in diazo derivatives. Chapters 5 and 6, which are entirely new, and of more interest to the organic chemist, are devoted to syntheses which can be achieved with diazoalkanes and diazoketones, and the mechanisms by which these and other reactions can be brought about. A forty-page chapter is concerned with reactions of aromatic diazo compounds other than the azo-coupling reaction. It is in this latter topic that Professor Zollinger excels, dedicating three chapters to a full description of the coupling reaction, the mechanism of azo coupling, and the application of the resultant theory to the production of azo dyes. The description of the author's own work, by which conclusive proof is given that the entering species in the azo-coupling reaction is the diazonium cation, may be regarded as a classical example and demonstrates admirably the opinion that these compounds must be examined from the physico-chemical aspect. The final chapter contains an account of the stability constants and stereochemistry of the metal complexes of some azo dyes, with an important introduction to the electronic orbital theory of bond-formation between dye and metal ion.

This book, which cannot be too strongly recommended to the serious student, could easily become a standard textbook, the only drawback being the demand on the student's pocket.

F. Jones

Pětijazyčný Slovník (Barvy, Laky, Povrchová, Úprava, Korose)

By R. Santholzer and J. Kořinský. Pp. 436. Prague: Státní Nakladatelství Technické Literatury. 1959. Price, Kčs. 20.20.

This dictionary covers very comprehensively the terms used in Czech, Russian, German, English, and French relating to the paint, varnish and lacquer industry, to surface treatment in general, and to corrosion. The first section of the book comprises a list of 2882 Czech terms, arranged in alphabetical order and numbered, together with the corresponding terms in the other languages. Subsequent sections contain alphabetical lists of the terms in each of the other languages in turn, together with the number under which the translations will be found in the first section. This dictionary is thus most easily used as an aid in reading or translating from Czech, but can be used without great difficulty for translating into Czech or for correlating the terms in any two of the five languages. The writer has, in fact, frequently used the 1956 edition for checking the precise meaning of French or German terms, experience having shown that it is more comprehensive and on the whole more accurate than most of the other multilingual dictionaries covering this field. The genders of all nouns and the accentuation of the Russian words are given. Explanatory notes are included where necessary to define the terms more closely.

The text of the new edition appears to differ from that of the previous one only in the inclusion of a list of abbreviations used in Russian— both editions contain a list of abbreviations used in the other languages, a useful set of tables correlating the units used in different countries or for different purposes (technical or scientific, large- or small-scale), and a list of journals, books, standard specifications, and dictionaries in the field of study and languages covered by the dictionary.

It is disappointing that no attempt has been made to fill in the few blanks left in the previous edition— surely it would have been possible in the three years that elapsed between the two editions to discover the French terms for the different types of paint brush or the Russian and French expressions for "filiform corrosion", "vehicle" or "binder", "smudge", and "feather edging" or "feathering", or if, as is suggested (though incorrectly in some cases), there are no single terms, to give some explanation of these simple ideas in each language. However, over 99% of the terms are given in all five languages.

D. R. Duncan

Surface Activity

By J. L. Moilliet, B. Collie, and W. Black. 2nd Edition. Pp. xvi + 518. London: E. & F. N. Spon Ltd. 1961. Price, 75s. 0d.

The first edition of this book (by the two first-named authors) appeared ten years ago. It was then very well received (J.S.D.C., **68** (1952) 25). But it is natural that, with such a rapidly developing subject, it had become out-of-date. The new edition is therefore very welcome. The general characteristic of the book remains the same— to combine considerable factual information with a very thorough theoretical background. It has been found possible to retain the same headings for the chapters as before, but these have been largely re-written and extended, the total increase being about 150 pages.

The three main parts deal with (I) the fundamental physical chemistry of surfaces and surface-active agents, (II) technical applications of synthetic surface-active agents, and (III) their chemical constitutions. In Part I, which occupies

some 200 pages, important new work is introduced on the states of solution of surface-active agents, on new experimental techniques for the study of adsorption, and on the theory of fundamental processes such as wetting, deflocculation, and protective colloid action. Part II, on technical applications, is both descriptive and explanatory, on the basis of the fundamental theory of Part I. Many new processes of technical importance are included. The aim is to relate the physical–chemical properties of the surface-active agent to the purpose of use, so that the reader should be able to choose surface-active agents for new purposes. Part III has had to be extended to deal with new types of surface-active agents. There is, of course, a continual change in the relative importance of the various types of compounds and mixtures of these, and the present edition reflects the present situation. References to the literature are given liberally in footnotes. A ten-page author index and a twelve-page subject index appear to be accurate and complete, and make reference easy.

The book is clearly written and gives the authoritative account of the subject that one would expect from the reputation and experience of the authors.

A. F. H. WARD

New Books Received

A.S.T.M. Standards on Textile Materials. A.S.T.M. COMMITTEE D-13 ON TEXTILE MATERIALS. Pp. xxii + 944. 1960. Philadelphia: American Society for Testing Materials. $9.75. Members $7.80.

Supplement to British Chemicals and their Manufacturers. 1960. London: Association of British Chemical Manufacturers.

Industrial Organic Nitrogen Compounds. MELVIN J. ASTLE. Pp. vii + 392. London: Chapman & Hall Ltd. 112s. 0d.

B.A.S.F. Bericht über das Geschäftsjahr. Pp. 45. Ludwigshafen-am-Rhein: B.A.S.F. 1961.

Dictionary of Dyeing and Textile Printing. H. BLACKSHAW and R. BRIGHTMAN. Pp. vi + 221. London: George Newnes Ltd. 1961. 30s. 0d.

The Report on the Census of Production for 1958. Part 88. Textile Finishing. BOARD OF TRADE. Pp. iv + 16. London: H.M.S.O. 1961. 2s. 6d.

Ciba. Annual Report 1960. Seventy-seventh Statement of Accounts of Ciba Limited, Basle. C.I.B.A. Pp. 43. Basle: Ciba Ltd.

The Dyeing of Cellulosic Fibres and Related Processes. S. R. COCKETT and K. A. HILTON. Pp. xiii + 416. London: Leonard Hill (Books) Ltd. 1961. 50s. 0d.

The Business of Management. ROGER FALK. Pp. 251. Harmondsworth: Penguin Books Ltd. 1961. 3s. 6d.

Telomerization and New Synthetic Materials. R. KH. FREIDLINA and SH. A. KARAPETYAN. Pp. x + 102. Oxford: Pergamon Press. 1961. 25s. 0d.

Patents, Designs and Trade Marks. Seventy-eighth Report of the Comptroller-General of Patents, Designs and Trade Marks, with Appendices for the year 1960. H.M.S.O. Pp. 27. London: H.M.S.O. 1961. 1s. 6d.

Pigmente. HANS KITTEL. Pp. xii + 824. Stuttgart: Wissenschaftliche Verlagsgesellschaft m.b.H. 1960. DM.178.

Colour and Colour Rendering of Tubular Fluorescent Lamps. A. A. KRUITHOF and J. L. OUWELTJES. Pp. 12. Reprinted from Philips Technical Review, Vol. 18. 1956–57.

Glyceryl Monostearate. The Composition, Properties and Uses of the Commercially Pure Material. E. S. LOWER. Pp. 11.

Fibre e Filati Artificiali e Sintetici. NOVACETA S.p.A. Pp. iii – 150. Milan: Novaceta S.p.A. 1960.

Paint Technology Manuals. Part I—Non-convertible Coatings. OIL AND COLOUR CHEMISTS' ASSOCIATION. (Hon. Ed. I. C. R. Bews). Pp. 326. London: Chapman & Hall Ltd. 1961. 30s. 0d.

Organic Coating Technology. Vol. II. Pigments and Pigmented Coatings. HENRY FLEMING PAYNE. Pp. viii + 624. London: John Wiley & Sons Ltd. 1961. 140s. 0d.

Scientific Thinking and Scientific Writing. MARTIN S. PETERSON. Pp. vii + 215. New York and London: Reinhold Publishing Corp. and Chapman & Hall Ltd. 1961. 56s. 0d.

Index of Chemistry Films. ROYAL INSTITUTE OF CHEMISTRY. (2nd edition). Pp. viii + 217. London: Royal Institute of Chemistry. 1961. 5s. 0d.

Register of Fellows and Associates 1960, with a Supplementary List of Graduate Members. ROYAL INSTITUTE OF CHEMISTRY. Pp. xx + 643. 1961.

Index des Huiles Sulfonées et Détergents Modernes. J. P. SISLEY. Pp. 560. Paris: Editions Teintex. 1960. 80 N.F.

Printing Ink Manual. SOCIETY OF BRITISH PRINTING INK MANUFACTURERS. Pp. viii + 746. Cambridge: W. Heffer & Sons Ltd. 1961. 70s. 0d.

1961 Buyers' Guide of Chemicals, Chemical Plant, Laboratory Equipment. THE SOCIETY OF CHEMICAL INDUSTRY. Pp. iv + 178. London: The Society of Chemical Industry. 1961. 7s. 6d.

Measurement of Absorbed Dose of Neutrons, and of Mixtures of Neutrons and Gamma Rays. Handbook 75. U.S. DEPARTMENT OF COMMERCE, NATIONAL BUREAU OF STANDARDS. Pp. 86. Washington: National Bureau of Standards. 1961. 35 c.

Units of Weight and Measure (United States Customary and Metric) Definitions and Tables of Equivalents. U.S. DEPARTMENT OF COMMERCE, NATIONAL BUREAU OF STANDARDS. Pp. 62. Washington: National Bureau of Standards. 1960. 40 c.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Technical Aspects of Function, Drive and Control in Development of the Cyclotric Jig

E. F. Rotter *Melliand Textilber.*, **42** (May 1961) 553–555

Precise maintenance of preset speeds and tension is essential in jig-dyeing. Technical aspects of function, drive, and control are discussed, considerations which led to the development of the Cyclotric jig. With this optimum process conditions can be maintained even under disadvantageous conditions. It is also satisfactory for the processing of structurally sensitive fabrics. Details of construction, etc., are given. I.G.L.

PATENTS

Drying Wet Carbon Black Pellets (C.I. Pigment Black 6 and 7)

Phillips Petroleum Co. *USP* 2,952,921 (26 Aug 1957)

Apparatus is described which dries the pellets in absence of O_2 and which uses little fuel. C.O.C.

Heat Treatment of Filaments or Yarns

Pye *BP* 870,061 (28 May 1958)

Apparatus is described for heating single filaments or yarns so that the heat can be rapidly terminated or controlled so as to obviate risk of overheating. C.O.C.

Yarn or like Material in Packages suitable for Treatment with Gas or Liquid

H. Buddecke *BP* 872,649 (Germany 16 Feb 1957)

A package of yarn, roving, or the like consists of a number of layers each wound in a number of equal flat coils arranged so that the upper run of one coil overlaps the lower run of the adjacent coil. The layers are arranged without interleaving so that the direction of the windings in one layer is at an angle (preferably 90°) to that of the windings in the adjacent layer. C.O.C.

Conveyor Belts for Textiles being treated with Aqueous Chlorites

Kunstzijdespinnerij Nyma *BP* 870,308 (Holland 6 May 1958)

Conveyor belts made of polyalkylene fibres are resistant to chlorite and so can be used for carrying textiles that are being treated with chlorite bleaching liquors. C.O.C.

Spraying Liquids onto Textile Materials

Walker & Smith, Batley *BP* 871,501 (23 Nov 1956)

Rinsing Yarns and Fabrics in Rope Form and in Continuous Lengths

Gebr. Stork & Co's Apparatenfabriek *BP* 873,642 (Holland 20 July 1957)

The goods pass over at least one winch which leads them through a bath of liquor beneath it. The rotation of the winch is reversed at regular intervals so that the goods pass through the liquor in a reciprocating manner. C.O.C.

Continuous Treatment of Textiles in an Autoclave

Kanegafuchi Spinning Co. *USP* 2,954,687 (3 May 1955)

The entry and exit to an autoclave are each pressure-reducing nozzles through which the textiles pass as tow or in rope form, the large surface-resistance of the textiles being used to prevent leakage from the autoclave. A valve may be used to control the internal area of the nozzle cross-section. Then, by the combined effects of the large surface-resistance to pressure leakage and control of the cross-sectional area of the passage, the textiles can be fed into and out of the autoclave continuously with no loss in pressure. Thus if 30–40% of the internal cross-sectional area of the nozzle is occupied by the sum of the cross-sectional areas of the filaments then, with a nozzle length of 5–10 mm, it is possible to work continuously at a cut-off pressure of 50–80 lb/in². C.O.C.

Taking Samples from Fabric in a Pressure-dyeing Machine

Maschinenfabrik Benniger *BP* 874,444 (Switzerland 27 July 1957)

Describes a device for taking a sample at any point across the width of the fabric without having to open the machine. C.O.C.

Guiding Travelling Webs

Pusey & Jones Corpn. *BP* 873,428 (U.S.A. 8 Nov 1956)

Maintaining Constant Tension in Travelling Webs

Witton-James *BP* 873,431 (12 Dec 1957)

Apparatus for Guiding Sheet Material over a Roller

Spooner Dryer & Engineering Co. *BP* 869,893 (3 Sept 1958)

Web Tensioning and Cleaning Apparatus

Oxy-Dry Sprayer Corpn. *USP* 2,956,300 (17 Sept 1956) *USP* 2,956,301 (12 July 1957)

Apparatus is described for applying controlled tension to paper, fabric, or plastic. It removes dirt, lint, etc. from the surface before the material is further processed, e.g. printed or coated. C.O.C.

Drying Roll

American Viscose Corpn. *USP* 2,956,348 (6 Aug 1956)

A temperature differential is maintained between the end and central surface portions of the roll. This has the advantage of ensuring that the edges of the film or sheet being treated are not dried more rapidly than the central portion of the sheet. C.O.C.

Drying Travelling Webs

E. A. Timson *BP* 872,003–4 (30 June 1958)

Burners employing liquid or gaseous fuel produce long and wide flat flames which issue lazily so that they are carried along and flow together and in contact with the web to be dried. The amount of heat applied is controlled in accordance with the speed of the web so as to prevent overheating and so that it can be safely built up progressively. C.O.C.

Irradiating Continuous Lengths of Material with High-energy Electrons

High Voltage Engineering Corpn. *BP* 872,518 (U.S.A. 28 Sept 1956)

Spray Damping Machines

Hunt & Moscrop *BP* 870,404 (19 Feb 1959)

In a mist-spray damping machine the spray from the nozzles impinges vertically downwards on to a pair of pivoted perforated spray boards. These boards convert the spray into a mist directed toward the fabric. The slope of the boards can be varied in order to control the volume of mist impinging on the fabric. C.O.C.

Apparatus for Impregnating or Coating Cloth with Polyvinyl Chloride, Rubber, or the like

Barrow, Hepburn & Gale *BP* 871,462 (12 May 1958)

The cloth is led through the impregnating material in a trough. Guide rollers are arranged in the trough so that each side of the cloth comes into contact with at least one roller. The cloth is led out of the bath upwardly between doctor blades to remove excess of coating material and between heating elements. Finally the cloth passes downwards between driving rollers placed at the discharge end of the apparatus. C.O.C.

Visibly Ascertaining the Position of the Weft in Travelling Cloth and Automatic Control of Weft-straightening Equipment

Heinz Mahlo *BP* 871,325 (Germany 26 Apr 1958)

Cuttling Mechanism

Fleissner & Co. *BP* 871,413 (Germany 30 May 1958)

Spreading Machine for Coating Fabric with Highly Viscous Material

Barrow, Hepburn & Gale *BP* 869,939 (15 Jan 1959)

When coating with a highly viscous material, e.g. pigmented polyvinyl chloride, the coating composition is drawn from a supply tank by a vacuum pump which delivers it into a trough having downwardly directed nozzles in its bottom. Interacting paddles in the trough force the composition through the nozzles on to fabric passing beneath it, the fabric then going to a device for spreading the composition over the fabric. C.O.C.

Spreading Powder or Granules over a Surface
C. van der Lely *BP* 872,604 (Holland 9 Nov 1956)
The apparatus has an oscillating ejector so controlled that it spreads the powder or granules uniformly.
C.O.C.

Dyeing Form for Producing Shaded Stockings
Munsingwear *USP* 2,953,915 (2 Apr 1958)

Yarn-waxing Device Readily Mounted Upon Any Machine
T. A. Purt and E. M. Bates *BP* 873,716 (27 Feb 1959)

Applying Wax or other Lubricants to Yarn During Winding (X p. 527)

II—WATER AND EFFLUENTS

Elimination of Aniline and Nitrobenzene from Production Wastes
K. Burdecki, J. Kucharski, and A. Sterninski
Przem. Chem., No. 12 (1959) 753–756
Polish Tech. Abs., No. 3/4 (39/40) (1960) 147–148
Adsorption on activated carbon is suitable for the recovery of aniline and nitrobenzene from waste waters and, with some modifications, for the recovery of aniline from other products. Experiments with Carbopol-Z-4 activated carbon gave the static sorptive power as 0·20 g/h and the dynamic as 0·10 g/h, but the use of columns with higher filling layer would raise the dynamic figure. The optimum rate of flow of waste through the column was found to be 1·5 m/h. R.A.

PATENT

Preventing Growth of Slime-forming Micro-organisms in Industrial Water
Shell International Research
BP 873,800 (U.S.A. 3 Apr 1959)
Addition of a compound of formula CH_2:CR·CHO (R = H or Alk of < 6 C), e.g. acrolein or methacrolein, prevents growth of slime-forming micro-organisms, particularly in the white water, i.e. water that has been previously used, employed in papermaking. C.O.C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Reactivities of Coordination Compounds. VI—Mechanism of Autoxidation of Iron (II) Chelates
T. Kaden and S. Fallab *Helv. chim. Acta*, 44 (May 1961) 714–720
Autoxidation of Fe^{2+} in the presence of the chelating agents 1,2-dihydroxybenzene-3,5- and 1,8-dihydroxynaphthalene-3,6-disulphonic acid is reported. That complexing agents with N-ligand atoms are without influence on the rate of the reaction $Fe^{2+} \rightarrow Fe^{3+} + e^-$ whereas with O-ligand atoms the rate of autoxidation is strongly increased, is explained by π-donor bonding in the case of Fe^{2+}–O, which increases electron density in the $Fe^{2+}d_\pi$-orbitals and thus favours the electron transfer to O_2 which is bonded to the metal ion by $d_\pi p_\pi$ overlap.
H.H.H.

Molan CN—Mothproofing Agent
M. Orzel *Prace. Inst. Włók.*, **9** (1959) 29–54
Chem. Abs., **55** (12 June 1961) 11862
Molan CN, a neutralised dihydroxypentachlorosulphotriphenylmethane, obtained by condensing 4-chloro-2-sulphobenzaldehyde with 2,4-dichlorophenol in presence of H_2SO_4, exhausts on to wool rapidly and levelly from a bath containing formic or sulphuric acid at 60°C.
C.O.C.

Pressure Cooking for Coating Colour Preparation
W. C. Black and V. L. Winfrey
Tappi, **44** (Feb 1961) 155–160
The degree of dispersion of the granules of a cooked starch paste is a function of time, temp., and shear. Reports that pressure cooking of a starch–clay coating improves viscosity were not confirmed, but clay dispersion was improved. Pressure cooking improves rub-resistance and overcomes the inhibiting effect of soap on starch gelatinisation. R.A.

PATENTS

Surfactants
Petrolite Corpn. *USP* 2,956,067 (5 July 1956)
The products obtained by combining a polyol (which must be susceptible to oxyalkylation) with epoxidised fatty-acid derivatives so as to introduce a fatty-acid radical by an ether linkage, followed by treatment with a polyamino derivative, are surfactants which are effective in both aqueous and non-aqueous liquids. C.O.C.

Antifoaming Agents
ICI *BP* 874,550 (5 Feb 1958)
Compounds of formula

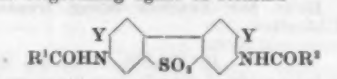
$$\begin{array}{l} R\text{–}COO(CH_2CH_2O)_mH \\ \quad\quad\quad\quad | \\ \quad\quad\quad O(CH_2CH_2O)_nH \end{array}$$

(R = residue of an estolide of ricinoleic acid or castor oil fatty acids; m and n = same or different whole numbers) are useful as antifoaming agents in aqueous media.
C.O.C.

Sequestering Agent
Joh. A. Benckiser Chemische Fabrik
BP 873,488 (Germany 19 Dec 1956)
Modification of *BP* 857,518 (J.S.D.C., **77** (May 1961) 219). A mixture of a condensed metal phosphate and nitrilotriacetic acid, $N(CH_2COOH)_3$, or a salt thereof, is a good sequestering agent for Ca, Mg, and Fe. C.O.C.

Sodium Zirconyl Silicate as Insolubilising Agent for Regenerated Protein Fibres
Courtaulds and F. W. Berk & Co.
BP 869,092 (21 July 1958)
Sodium zirconyl silicate is an outstanding insolubilising agent for use in the manufacture of regenerated protein fibres. C.O.C.

Bleaching Composition containing a Dibenzothiophen Dioxide Fluorescent Brightening Agent
American Cyanamid Co. *BP* 872,553 (28 July 1958)
A bleaching composition for cellulosic fibres consists of a solid bleaching agent, e.g. bleaching powder, and a fluorescent brightening agent of formula



(R^1CO and R^2CO = aromatic carboxylic acyl radicals; Y = H or $NaSO_3$; R^1 and/or R^2 contains SO_3H when Y = H) e.g. the compounds described in *BP* 684,842 and 696,038 (J.S.D.C., **69** (1953) 95, 465) in such proportions that the composition contains > 2% available chlorine.
C.O.C.

Stabilising Bleaching Compositions containing Dichlorocyanuric Acid and its Salts
Unilever *BP* 872,815 (13 Aug 1959)
Mixtures of dichlorocyanuric acid and its salts with synthetic detergents often gradually deteriorate in bleaching power, especially when moisture is present. This loss in bleaching power is much reduced if the dichlorocyanuric acid is present not as a fine powder but as granules of which 70% are retained on a 60-mesh B.S. sieve. C.O.C.

Ampholytic Compositions for the Wet Treatment of Textiles
General Aniline *USP* 2,953,526 (20 Dec 1955)
Mixtures of an ampholytic compound of formula $R^1R^2N\cdot R^3COOH$ (R^1 = Alk of > 7 C; R^2 = H, Alk, hydroxyalkyl, hydroxyalkoxyalkyl, or hydroxypolyalkoxyalkyl; R^3 = alkylene of 1–4 C) and 4–300% of its wt. of a compound of formula $R^4(CH_2CH_2O)_nH$ (R^4 = alkoxy or alkylphenoxy; n > 4) are stable to acids, alkalis, and hard water. They have good wetting, dispersing, and detergent properties, are not salted out by large concentrations of electrolytes, and are compatible with resin finishes. They have very wide uses as softening agents, lubricants, detergents, etc. C.O.C.

Non-ionic Detergent Composition imparting an Antistatic Finish to Nylon
British Nylon Spinners *BP* 873,214 (20 Aug 1958)
A mixture of a non-ionic surfactant (51·0–98·5%), a cationic surfactant (1·0–48·5%), and an anionic surfactant (0·5–24·0%), in which the cationic surfactant

is present in stoichiometrical excess over the anionic surfactant, are excellent detergents for nylon, to which they impart an antistatic finish which is not removed by rinsing. C.O.C.

Sizing Fibrous Cellulosic Materials
Kerr-McGee Oil Industries *USP* 2,954,313 (17 Oct 1955)

A size which can be used under alkaline or acid conditions consists of an aqueous emulsion of a "bitumen" prepared by use of an organic emulsifying agent and in which $\leqslant 95\%$ of the bitumen particles have diameter 0·1–1·0 μ, $\leqslant 99\%$ are $< 4\,\mu$ and none are $> 6\,\mu$. The bitumens particularly claimed are the heavy mineral oils having lubricating properties. C.O.C.

Lubricants for Cellulose Ester Fibres
Eastman Kodak Co. *USP* 2,956,949 (19 Oct 1955)

Effective lubrication and antistatic properties are obtained by use of a mixture of (*a*) mineral oil of 50–150 s Saybolt Universal viscosity at 100°F (10–80 parts by wt.), (*b*) aliphatic alkyl acid phosphate (4–20), part of which may be replaced by an alkyl sulphate, (*c*) alkylolamine (1–10), (*d*) long-chain aliphatic acid (1–15), (*e*) sulphonated oil or fat (4–50), (*f*) a glycol ether (1–15), and (*g*) a tartrate (4–15). The last four components also confer on the mixture excellent sizing properties. C.O.C.

Size for Nylon
Monsanto Chemical Co. *BP* 871,589 (4 Sept 1958)

Water-soluble propylene–maleic interpolymers containing recurring units of formula

$$\left[CH_2{\cdot}CH(CH_3){\cdot}CH{\cdot}CH(COOY){\cdot}CH(COOY)\right]$$

(one Y = H, the other = H or is derived from an alkanol) and of specific viscosity $\leqslant 0{\cdot}1$ as a 1% solution in dimethylformamide at 25°C when both Ys = H or after partial esterification when one Y is derived from an alkanol, are excellent sizing agents for nylon, from which they are readily removed during scouring. C.O.C.

Softening Agents and Textile Lubricants having Surface-active Properties
Atlas Powder Co. *BP* 872,106 (U.S.A. 23 Apr 1957)

A 3-halogeno-2-hydroxypropyl ether of a polyhydric alcohol (produced by condensing 1 mol. of a polyhydric alcohol having 3–6 OH groups with $\leqslant 1$ mol. epihalogenohydrin) is condensed with an amide, imidazoline, or tetrahydropyrimidine containing basic N (produced by condensing 1 mol. of a polyalkylene polyamine, containing 3–5 amino N atoms and 2–3 C atoms in each alkylene group, with $\leqslant 1$ mol. of a non-aromatic monocarboxylic acid of 12–22 C or aliphatic monocarboxylic acid of 2–6 C) in the proportion of 1 halogen equivalent of the former to 1–3 amino equivalents of the latter at 75–150°C, to yield very viscous syrups having surface-active properties. C.O.C.

Anhydrous Oil-base Lubricating and Softening Composition for Hydrophobic Cellulose Derivative Fibres
American Viscose Corpn. *USP* 2,956,950 (12 Feb 1958)

N-Hydrocarbon-trimethylenediamine dioleate (hydrocarbon is a satd. or unsatd. straight chain of 14–18 C) is a highly effective coupling agent for a water-soluble or water-dispersible antistatic agent and mineral oil in a fibre lubricant. It is insoluble and non-dispersible in water, so that it is necessary to use an emulsifying agent to render the mixture of antistatic agent, mineral oil, and the above compound readily removable by scouring. C.O.C.

Aminoplast Precondensates
Courtaulds *BP* 872,656 (U.S.A. 4 June 1957)

Modification of *BP* 796,815 (J.S.D.C., **74** (1958) 659), better results being obtained by using an alkaline-earth hydroxide, preferably $Mg(OH)_2$, instead of NaOH. The resulting precondensates cure more readily than those made in presence of NaOH. C.O.C.

Aqueous Emulsion Copolymers as Thickening Agents
Rohm & Haas Co. *BP* 870,994 (U.S.A. 21 Nov 1956)

A 25–50% aqueous dispersion of a water-insoluble copolymer of methacrylic acid (25–70% by wt.), C_1–C_4 alcohol acrylates ($\leqslant 10$), and one or more neutral monoethylenically unsaturated monomers (0–40) is a mobile liquid useful for direct use as a thickener for both aqueous and organic-solvent systems, especially for those to be applied to porous substrates, e.g. textile printing pastes and backfilling sizes. C.O.C.

Wax Emulsions for Imparting Water Repellency
BASF *BP* 869,961 (Germany 19 Oct 1957)

Emulsions of paraffin wax, fatty acids, and/or resin acids containing in an aqueous acid disperse phase di-, tri-, or tetra-valent weakly basic metals but no protective colloid and, as sole surface-active emulsifying agent, 2–20% on wt. of the emulsion of an alcohol of < 6 C, are very stable, e.g. they show no separation on being centrifuged or when subjected to repeated alternations of temperature. C.O.C.

Water-repellent Finish with Wax and Titanium or Zirconium Compounds
Catomance *BP* 869,952 (10 Dec 1957)

A neutral or alkaline aqueous emulsion of a wax is used which contains dissolved in the wax a water-insoluble organic Ti or Zr compound in which the metal is tetravalent and in which each of these valencies is directly attached to an alkoxy, hydroxyalkoxy, or acyloxy radical or a condensed form of such compounds in which three valencies are so attached and the fourth is attached to an O bridge between two metal atoms, e.g. a dispersion of a 1% solution of 2-ethylhexyl titanate in paraffin wax. C.O.C.

Water-repellent Finish for Water-absorbent Materials
Petren Products *BP* 870,397 (U.S.A. 3 Oct 1956)

The aqueous solutions obtained by mixing neutral aqueous solutions of potassium alum (32 by wt.) and lead acetate (48) and removing the precipitated $PbSO_4$ are used to render paper and other fibrous materials water-repellent, by simple impregnation and drying. C.O.C.

Werner-type Aluminium Complexes— Water-repellent Agents and Sizes
DuP *BP* 869,951 (6 June 1957)

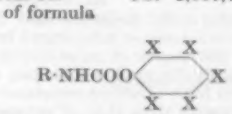
Werner-type complexes comprising positive complex ions in which nuclear Al atoms are covalently linked to O atoms in OH groups and/or O atoms linked to other Al atoms, the number of O to Al atoms being $> 1:1$ but $\geqslant 2{\cdot}5:1$ are co-ordinated with the acyloxy groups of carboxylic acids of solubility in water $< 1\%$ by wt. at 20°C and as negative ions the anions of strong inorganic acids, e.g. stearato aluminium chloride or aluminium–palmitic or –myristic acid complexes, are used to impart water-repellency to and to size paper and cellulosic and synthetic-polymer fibres. C.O.C.

Perfluoro-substituted Aliphatic Acids and their Derivatives— Oleo- and Hydro-phobic Agents
Minnesota Mining & Manufacturing Co. *USP* 2,951,051 (26 June 1957)

Acids of formula $R^1CH_2{\cdot}R^2{\cdot}CH_2COOH$ (R^1 = monovalent, functionally unsubstituted perfluorinated group of 4–12 C; R^2 = straight-chain alkylene of 1–12 C) and their derivatives have low acid strength and exhibit the full oleophobic properties of the perfluorinated group, e.g. 11-(perfluoro-4-ethylcyclohexyl)hendecanoic acid, poly-(vinyl 11-(perfluoro-octyl)-hendecanoate) or a copolymer of vinyl 11-(perfluoro-octyl)-hendecanoate with maleic anhydride, are used to impart an oleo- and hydro-phobic finish to textiles. C.O.C.

Rotproofing Agents
Spencer Chemical Co. *USP* 2,951,786 (14 Jan 1955)

Compounds of formula



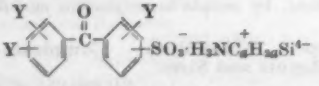
(X = Hal; R = phenyl or nuclear-substituted phenyl) e.g. pentachlorophenyl-*N*-(bromophenyl) carbamate, are highly effective rotproofing agents having very good fastness to leaching. They are colourless and are applied from solution in organic solvents or as aqueous dispersions. C.O.C.

Protective Agents Against Termites
FBy *BP* 873,590 (Germany 10 July 1959)
Compounds of formula $R^1{\cdot}SO_2{\cdot}NHR^2$ (R^1 = CH_3 or C_6H_4 substituted by 1–3 Cl; R^2 = subst. or unsubst. benzene or naphthalene) give good protection to textiles, paper, leather, rubber, wood, plastics, etc. against attack by termites, presence of 1–2% being effective. Thus, filter paper 0·5 mm thick and 9 cm in diameter was evenly impregnated with 2 ml of a 0·05% acetone solution of chloromethylsulphonyl-2,4-dichloroaniline, air-dried for 4 days, and then exposed to 20 worker termites. All the insects were dead after 24 h and the paper was undamaged. Tests with a 0·005% solution gave the same result. After being kept in a tropical climate for several months the paper treated with the 0·05% solution still showed the same good proof against termites. C.O.C.

Rot- and Insect-proofing Agent for Cellulosic Materials
Pan American Petroleum Corpn.
USP 2,951,789 (27 Dec 1955)
The heavy-metal soap of the acid mixtures produced in hydrocarbon synthesis, particularly those of acids of $C > 12$, are excellent preservatives for cellulosic textiles, wood, etc. Their durability is greater than that of the heavy-metal soaps hitherto used for this purpose. C.O.C.

Bacterio- and Fungi-static Agents
Monsanto Chemicals *BP* 873,602 (19 Dec 1957)
2-Mercapto benzothiazoles having > 1 Hal substituted in the benzene ring, two of the Hal being *ortho* to each other, have excellent antiseptic properties when present in plastics, rubber, wax polishes, soaps, etc. C.O.C.

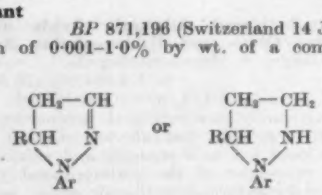
Absorbers of Ultraviolet Radiation
Union Carbide Corpn. *USP* 2,950,986 (22 Aug 1957)
Compounds of formula



(Y = H, OH, Alk, alkoxy, nitro, or dialkylamino; $a > 2$) are excellent absorbers of ultraviolet radiation. C.O.C.

Light Stabiliser for Halogeno Resins
Monsanto Chemical Co. *USP* 2,951,052 (31 Mar 1958)
A mixture of a compound of formula $R^1OP(OR^2)OR^3$ (R^1 and R^2 = H or organic radical of 4–18 C; R^3 = organic radical of 4–18 C), e.g. triphenyl phosphite, and a 2-hydroxybenzophenone having an absorption of 85% in the 340–400 mμ region, protect halogeno resins against discoloration and stiffening on exposure to heat and/or outdoor weathering. C.O.C.

Antioxidant
Gy *BP* 871,196 (Switzerland 14 June 1957)
Addition of 0·001–1·0% by wt. of a compound of formula



(R = H or Alk), e.g. 1-phenyl-pyrazoline or -pyrazolidine, very effectively prevents oxidation of organic compounds. C.O.C.

Water-dispersible, Partially Substituted Derivatives of Amylose
National Sugar & Starch Corpn.
BP 871,634 (14 Aug 1959)
Amylose is molecularly dispersed and then esterified or etherified so as to introduce substituent branches on the molecule. When the degree of substitution is 0·02–0·15, the product is readily dispersible in water, and films cast from the dispersion have excellent resistance to water and tensile strength greater than that of comparable films of regenerated cellulose or cellulose acetate. C.O.C.

Binding Agent for Fixing Pigments on Fibrous Material
FH *BP* 873,318 (Germany 26 Sept 1957)
An aqueous binding agent consists of the alkali-soluble condensate containing reactive carboxylic groups obtained by reacting a polycarboxylic acid with a polyhydric aliphatic alcohol, and a water-soluble precondensate of phenol, urea or a derivative thereof, or melamine, and an aldehyde. C.O.C.

Binding Agents for the Flock-coating of Textiles
FBy *BP* 872,525 (Germany 30 Nov 1956)
Aqueous emulsions containing both *N*-methylol polyethers and film-forming polymers containing methylol or methylol–alkylether groups and amide, hydroxy, or ester groups, when used as binders in flock-coating, yield products having a soft handle and excellent fastness to washing, rubbing, and organic solvents. C.O.C.

Stable Aqueous Emulsions of Polymers which can be Cross-linked by Oxidation
FBy *BP* 870,015 (Germany 16 Apr 1958)
The emulsions are obtained by emulsion polymerisation of 2–50% of an ester of an αβ-ethylenically unsaturated carboxylic acid and a saturated aliphatic or cycloaliphatic alcohol containing one hydroxyl group and at least one ether oxygen atom with 98–50% of an ethylenically unsaturated monomer in contact with a redox catalyst system. The catalyst system comprises a free-radical-forming catalyst in which there is molar excess of a reducing agent. Fibrous materials coated with these emulsions have excellent resistance to heat and water, extremely low permeability to steam, and retain their handle. Used as pigment binders for leather finishes they have exceptional thermal stability and are absolutely resistant to hot ironing. C.O.C.

Driers (V p. 517)
Flame-resistant Cellulosic Compositions (VI p. 519)
Binding Agents for Pigment Dyeing or Printing (IX p. 524)
Catalyst for Aminoplast Finishes (X p. 527)
Aqueous Dispersions of Nylon (XIII p. 532).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Energetic Condition of Excited States in Molecules and Dyes and its Relation to Chemical and Photochemical Properties
G. Scheibe *Chimia*, **15** (Jan 1961) 10–20
A brief survey of previous work in this field precedes a discussion in electronic terms of the absorption spectra of a protonated ψ-isocyanine, an *N*-methylquinaldinium cation, *cis*- and *trans*-forms of a dipyrrolidylpentamethine cation, some simple anionic and cationic dyes, cpd. of the 2,2- and 2,4-diquinocyanine type, a dibenzopyrrylium-ψ-isocyanine, and a 2,2-diquinolylmethane–zinc chloride complex. Protonation enthalpies and photoexcitation energies of the cyanine dyes are dealt with, and a schematic arrangement of the energy levels of sensitisers and desensitisers relative to the valency and conductivity in silver bromide is included. 29 References. H.H.H.

Methods for the Arrangement of Absorption Bands of Dyes for Calculated Transitions
H. Labhart *Chimia*, **15** (Jan 1961) 20–26
A survey is first made of the most important methods available (absolute, electrically induced dichroism, relative, photo-dichroism, and fluorescence polarisation) for the determination of the polarisation direction of absorption bands. Then the electrically induced dichroism method is given theoretical and experimental description, and is applied to anthraquinone deriv. The results obtained are compared with the theoretical LCAO-MO data of Kuboyama (*Bull. Chem. Soc. Japan*, **31** (1958) 752) and the four possible transitions considered. Substitution by chlorine only slightly influences the energy values. H.H.H.

Analogy Considerations and Calculations for the Quantum Mechanical Treatment of Light Absorption by Dyes
H. Kuhn *Chimia*, **15** (Jan 1961) 53–62
The wave-mechanical treatment of chain dyes is reviewed, the cases of branched electronic systems and of hetero-atoms are considered, and the course of the potential along the chain is worked out. Finally the Coulomb interaction between the photo-electrons and the other electrons is dealt with. H.H.H.

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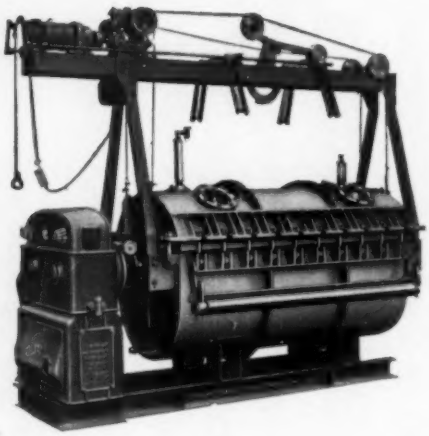

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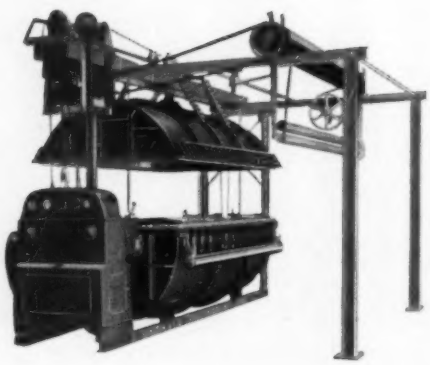

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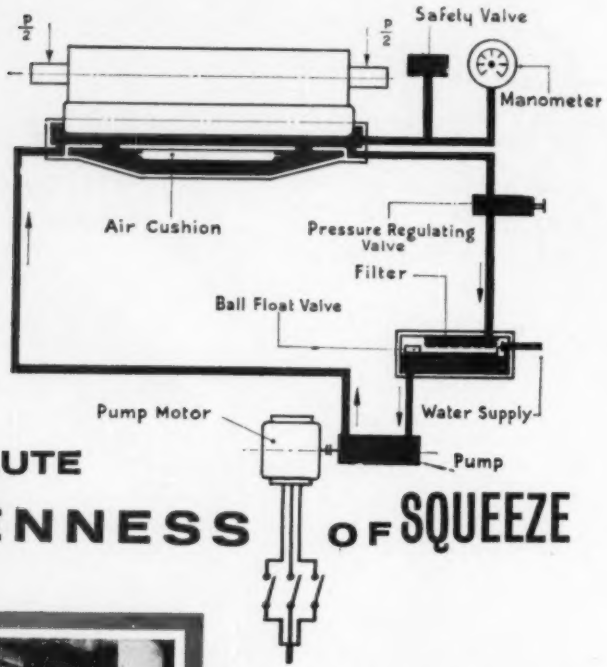

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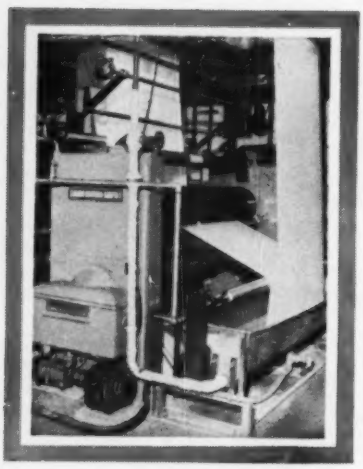

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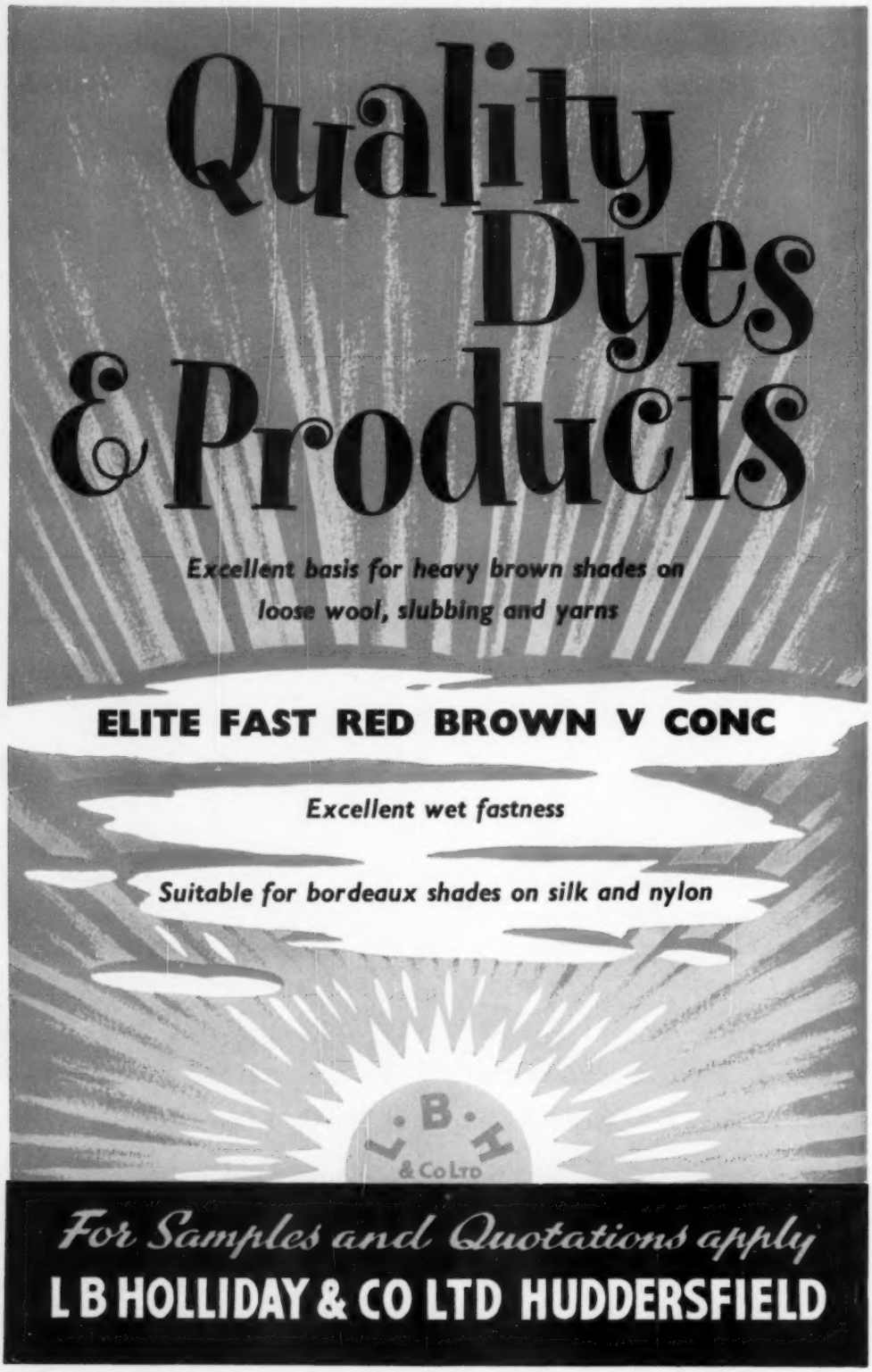
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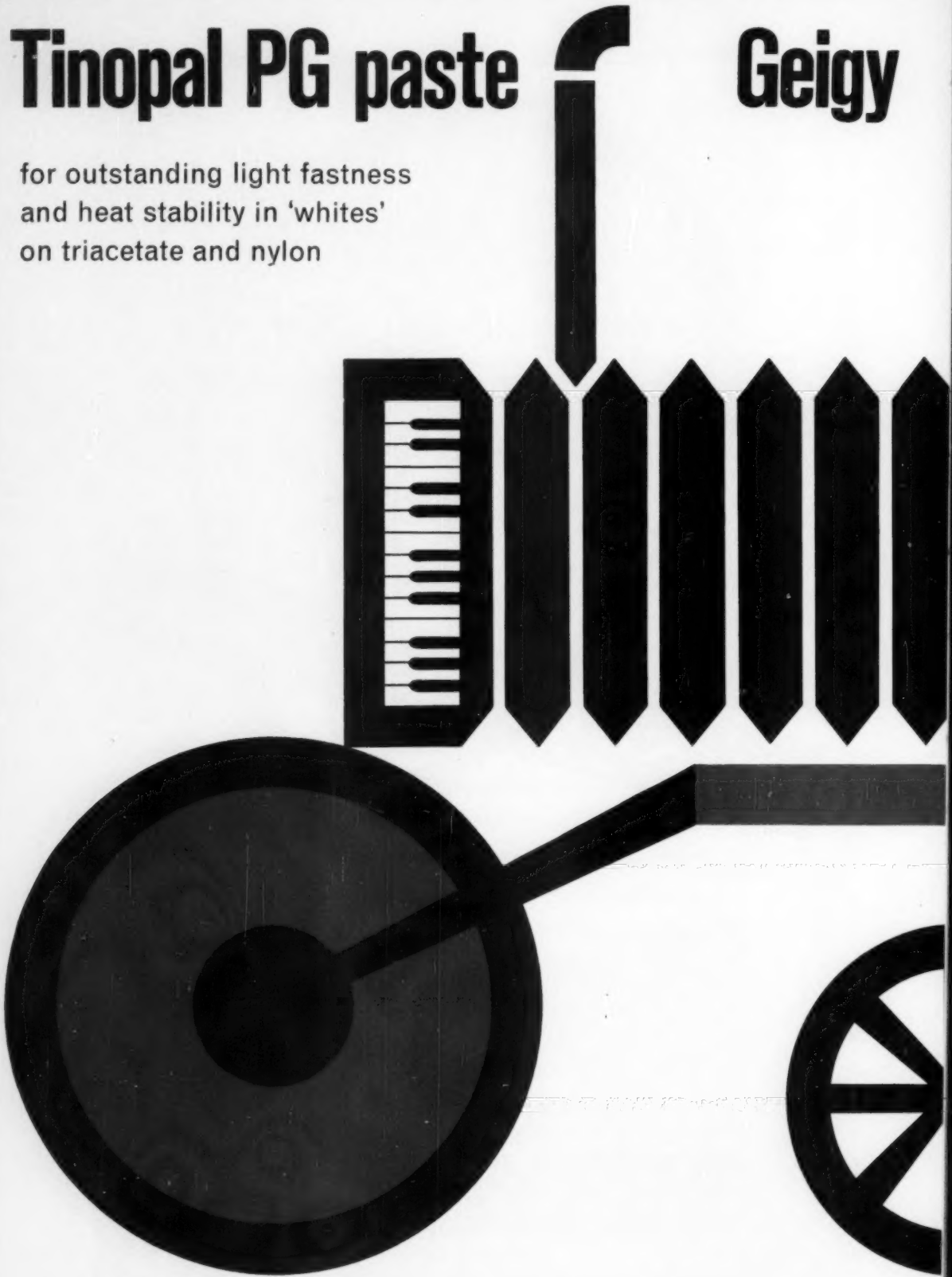

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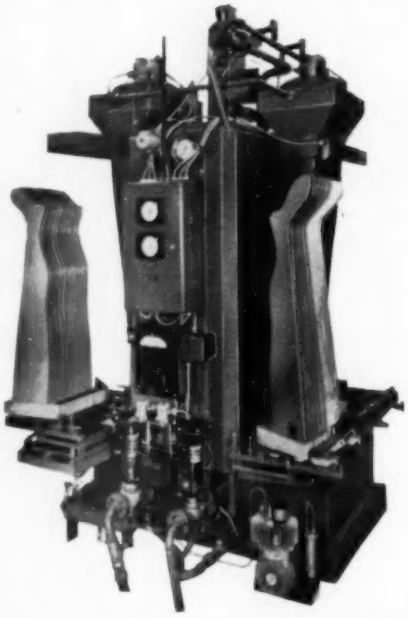

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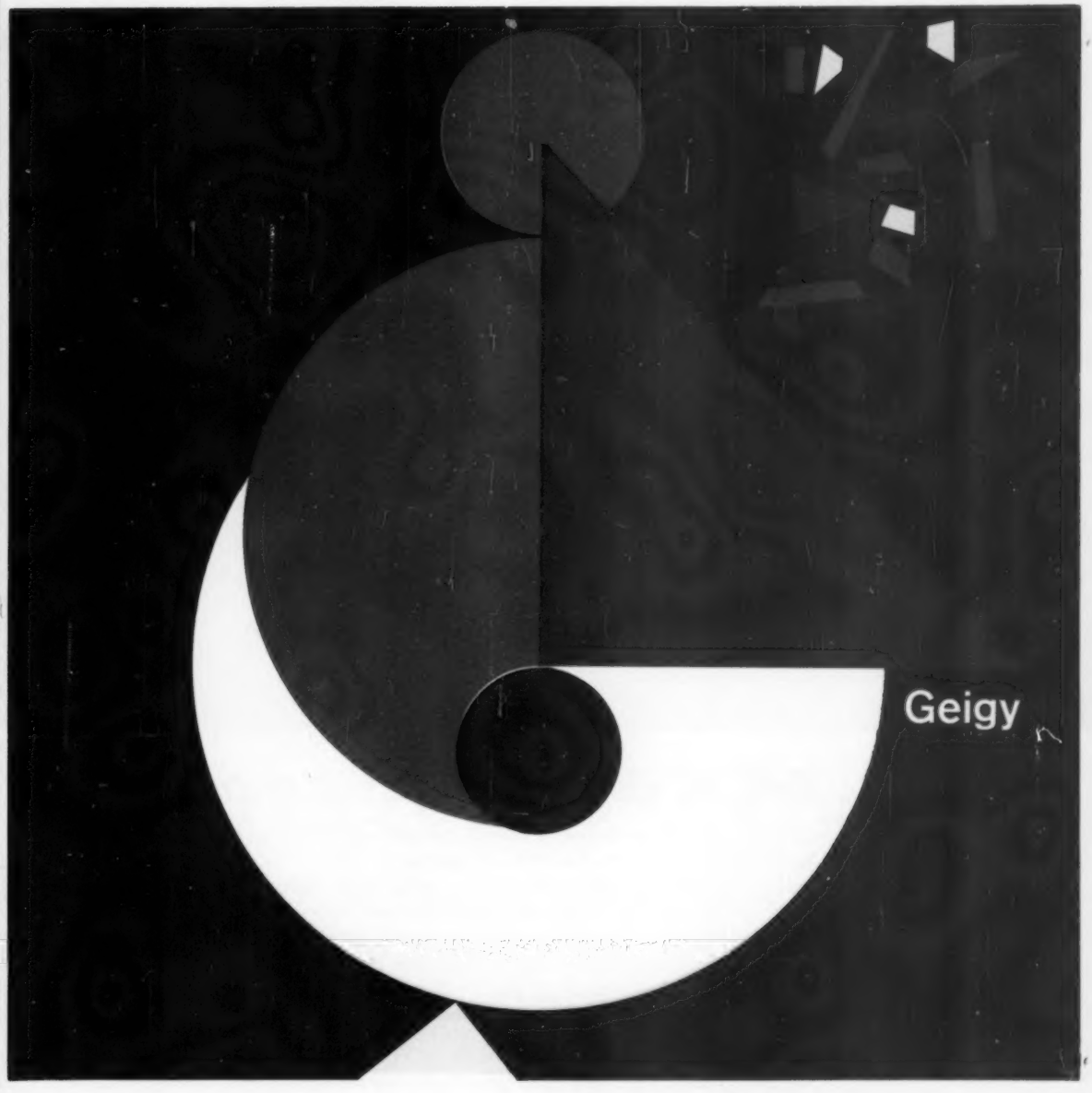

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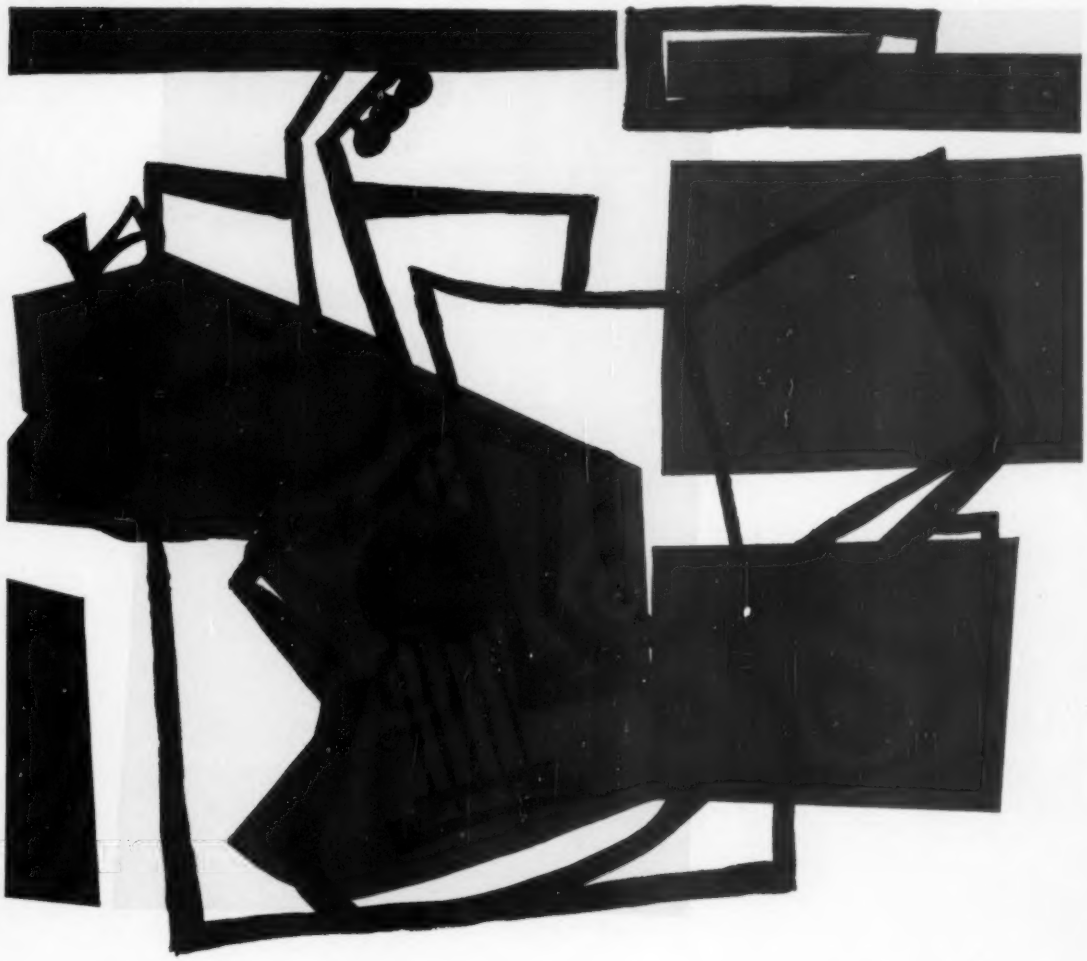

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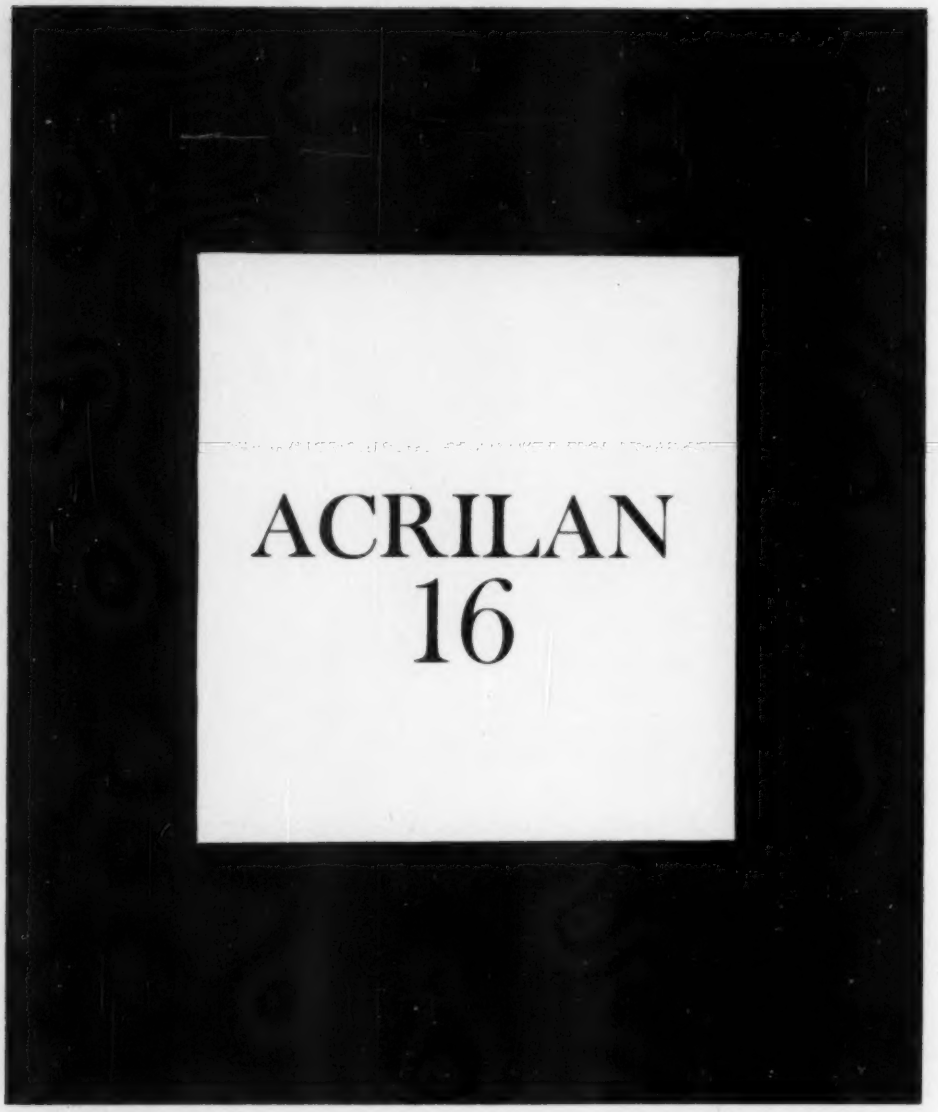

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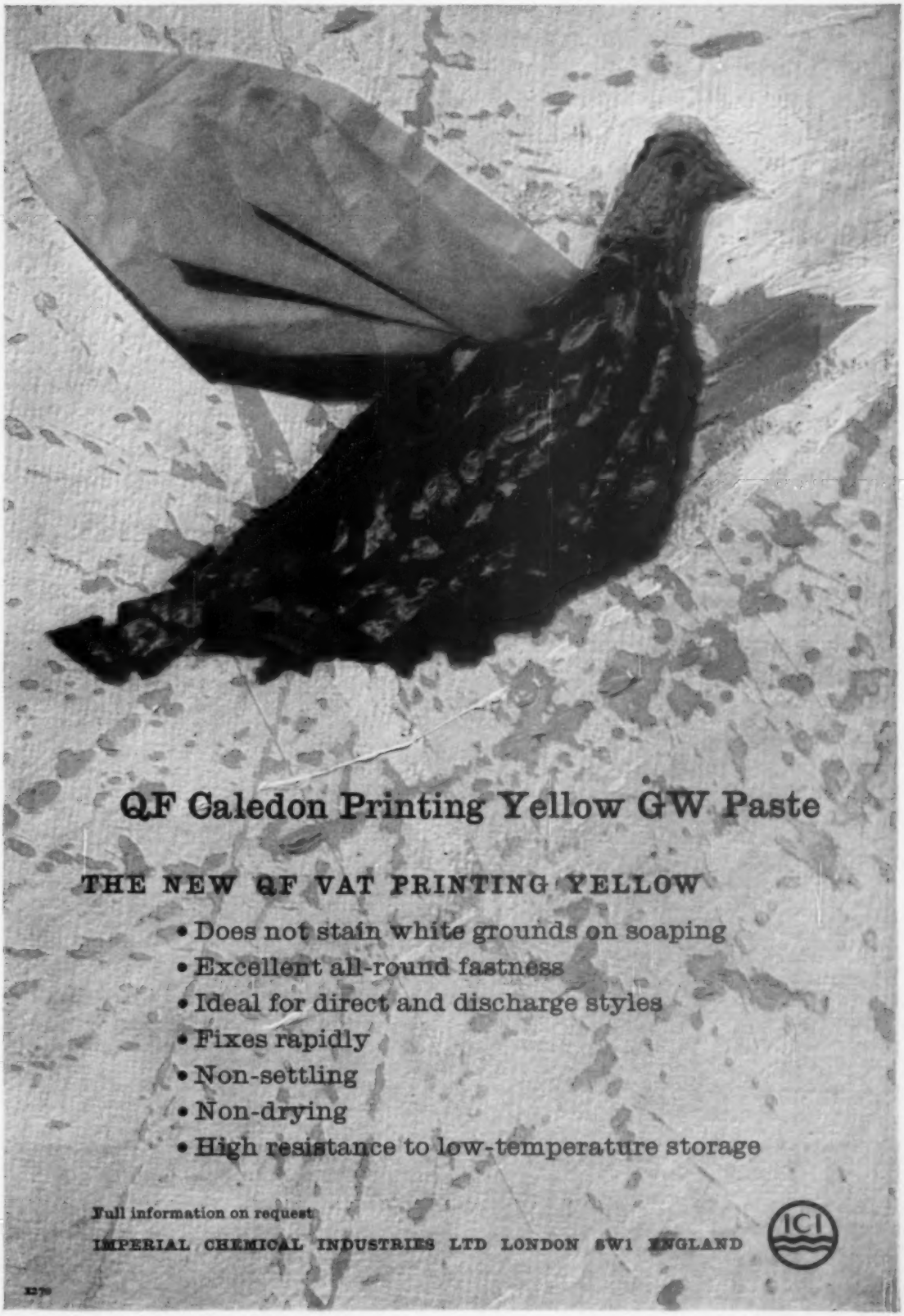
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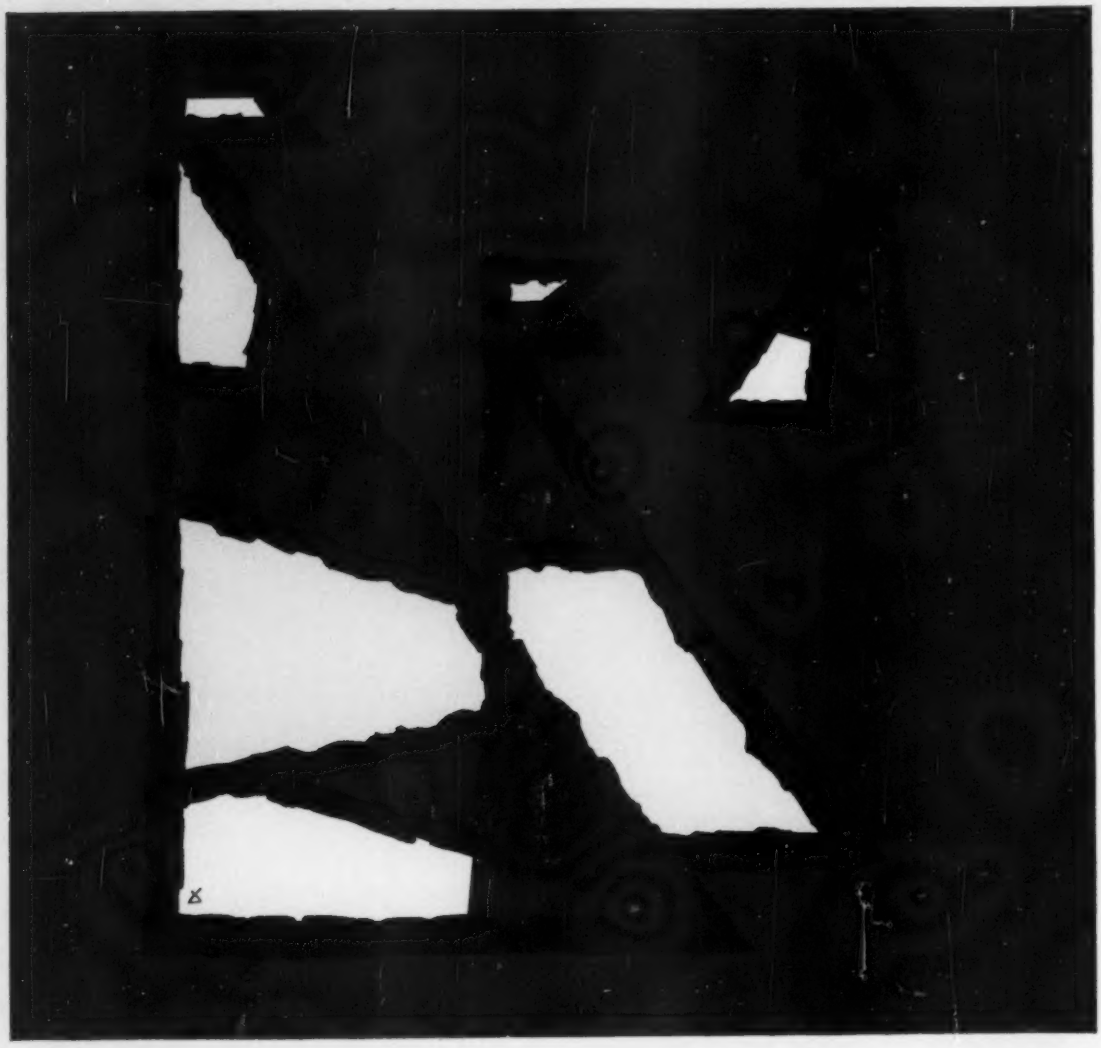

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The Inversion of the Auxochrome
R. Wizinger *Chimia*, **15** (Jan 1961) 89–105

General discussion with reference to coloured compounds of many chemical types. It is claimed that auxochrome inversion has a much greater significance in the relation between colour and constitution than has hitherto been accepted. A.T.P.

Kinetics of the Reaction of Cyanuric Chloride with Aniline and Monomethylaniline in a Non-polar Medium
B. Bitter and H. Zollinger *Helv. Chim. Acta*, **44** (May 1961) 812–823

Reaction rates in benzene soln. are measured for (1) cyanuric chloride and aniline, (2) 2-anilino-3,5-dichlorotriazine and aniline, and (3) 1-*N*-methylanilino-3,5-dichlorotriazine and *N*-methylaniline. Reaction (1) is autocatalytic, of first-order with respect to cyanuric chloride and approx. so with respect to aniline (weak base catalysis by aniline); (2) is also autocatalytic, of second order with respect to 1-anilino-3,5-dichlorotriazine and approx. so to aniline; (3) is not autocatalytic. All reactions are catalysed by triethylamine and by pyridine, as well as by bifunctional catalysts, e.g. carboxylic acids and α-pyridone, but not by γ-pyridone or phenols. H.H.H.

Purity of Food Dyes
L. Piekarski and S. Krauze *Roczniki Państwowego Zakładu Hig.*, **11** (1960) 551–558 *Chem. Abs.*, **55** (26 June 1961) 12687

Analysis by ascending paper chromatography with butanol, ethanol, and water (2:1:1) as solvents revealed that many commercial food dyes, both Polish and imported, contain a large number of impurities, indicating the need for better quality control. C.O.C.

Pigments and Dyes for Plastics
G. M. Reiner

I *Pitture e vernici*, **36** (1960) 789–797

A detailed examination of colorants for plastics, with special attention to their dispersibility, brilliance, and hiding power, and fastness to migration, light, weathering, and heat.

II *Ibid.*, 897–906

A list of colorants (and their characteristics) suitable for use with nitrocellulose, cellulose acetate, cellulose acetate butyrate, hardened casein, phenolic resins, and aminoplasts.

III *Ibid.*, 1085–1100 *Chem. Abs.* (**55** (10 July 1961) 13895

A similar list of colorants suitable for polymethyl methacrylate, polyethylene, polystyrene, polyester resins, polytetrafluoroethylene, polyvinyl chloride, polyamide resins, and natural and synthetic rubbers. C.O.C.

New Azo Dyes derived from Compounds containing a Reactive Methyl Group
H. Wahl *Chimia*, **15** (Jan 1961) 126–133

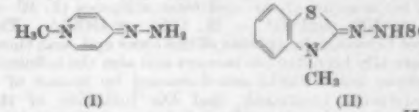
The coupling reactions of mono- and bis-diazonium salts with reactive cpd., e.g. methylbenzothiazole and dimethyl-2,3-benzimidazole, to give mono-azo and dis-azo dyes are described. The special cases of trimethyl-2,3,3-indolenine, methylbenzoxazole, and the double coupling on acetanilidovinyl and anilinovinyl deriv. are then considered. Finally, the mechanism and kinetics of these coupling reactions are discussed. H.H.H.

Infrared Spectra and Structure of Arylazonaphthols
K. J. Morgan *J.C.S.*, (May 1961) 2151–2159

Infrared spectra indicate the presence of an azo–hydrazone tautomerism both in the solids and in the soln. of 1-arylazo-2- and 4-arylazo-1-naphthols. The aryl-portion of both series of naphthols is monosubstituted by the following: H and *o*-, *m*-, *p*-OCH_3, CH_3, Cl, and NO_2 respectively. The position of equilibrium moves towards the hydrazone in polar solvents and under the influence of electron-withdrawing substituents in the aryl group; the hydrazone content increases in the order: $p\text{-}OCH_3 < o\text{-}CH_3 \simeq p\text{-}CH_3 < m\text{-}CH_3 < H < m\text{-}OCH_3 < m\text{-}Cl < m\text{-}NO_2 < o\text{-}OCH_3 \simeq o\text{-}NO_2$. H.H.H.

New Routes in Azo Chemistry
S. Hünig *Chimia*, **15** (Jan 1961) 133–137

Previously published methods (cf. *Angew. Chem.*, **70** (1958) 215) for the preparation of heterocyclic azo dyes which are inaccessible by direct coupling, are now supplemented by a process based on the oxidative coupling of cpd. which can readily form the amidrazone system, $>\!=N\text{–}NH_2$, with phenols, reactive methylene cpd., and aromatic amines. Examples of such cpd. are I and II below. Oxidative couplings by asymmetric diaryl-hydrazines are described together with the chemistry of dye formation and the role of the super-reactive diarylhydroxylamines.



(I) (II)

H.H.H.

Synthesis of Chlorantine Fast Violet 2RLL
T. Nishi, Y. Abe, and S. Ouchi *Kogyo Kagaku Zasshi*, **61** (1958) 790–792 *Chem. Abs.*, **55** (29 May 1961) 10466

Coupling of J acid in presence of soda ash with diazotised 2-aminophenol-4-sulphonic acid or 2-aminoanisole-4-sulphonic acid, condensation of cyanuric chloride with the resulting aminomonoazo compound, and formation of the Cu-complexes are described. [Abstractor's note—The dye of this name is given in the *Colour Index* as C.I. 29225 with a structure based on urea-di-J acid and having no cyanuric ring.] E.S.

Chlorotriazinyl Dyes
K. Higashiura *Technol. Repts. Kansai Univ.*, **32** (1960) 133–138 *Chem. Abs.*, **55** (12 June 1961) 11853

The colours are recorded of cold-dyeing reactive dyes made by condensing 1 mol. of cyanuric chloride (I) with 1 mol. of amines and aminoazo compounds, and of hot-dyeing ones from 1 mol. of I and 2 mol. of the same or different amines and aminoazo compounds. E.S.

Studies on Several Reactive Dyes

II
T. Kitao, N. Kuroki, and K. Konishi *Bull. Univ. Osaka Prefecture*, Ser. A **8**, No. 1 (1959) 57–69

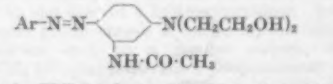
Reactive dyes containing 3-chloro-2-hydroxypropyl, α,β-dichloropropionyl, or aldehyde groups were prepared by condensing epichlorohydrin or α-β-dichloropropionyl chloride with the NH_2 group of suitable water-soluble dyes, and by coupling diazotised aminobenzaldehydes with pyrazolone- or naphthol-sulphonic acids, and examined by dyeing on silk, cellulose, and synthetic fibres.

III
T. Maruyama, T. Kitao, N. Kuroki, and K. Konishi *Ibid.*, Ser. A **8**, No. 2 (1960) 77–92 *Chem. Abs.*, **55** (12 June 1961) 11854

The F atom in soluble and disperse dyes containing an SO_2F group is labile and enables such dyes to combine chemically with various fibres in presence of mild alkali. E.S.

Synthesis of Benzimidazole Compounds containing Bis(β-Chloroethyl)amino Groups—II
O. F. Ginzburg, B. A. Porai-Koshits, and K. Yu. Maryanovskaya *Zhur. obshch. khim.*, **30** (Feb 1960) 570–573

Reduction of aryl derivatives of *o*-aminoazo dyes of the type



with $PbCl_2$ in HCl leads to the formation of imidazole deriv. This method was used for the preparation of 2-methyl-5(6)-bis(β-hydroxyethyl)amino-benzimidazole, which with thionyl chloride was converted to 2-methyl-5(6)-bis(β-chloroethyl)aminobenzimidazole. T.Z.W.

Improving the Quality of Direct Dyes
L. Légrádi and T. Kovács *Magyar Kém. Folyóirat*, **67** (1961) 1–3 *Chem. Abs.*, **55** (26 June 1961) 12857

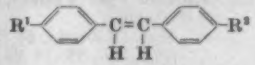
Coupling Bismarck Brown (C.I. 21000) with 1-chloro-2-amino-4-benzenesulphonic acid, instead of sulphanilic acid (as used to prepare Dianil Brown, C.I. 35005), yielded a dye of better light fastness but otherwise of the same fastness properties. 1-Chloro-2-amino-4-benzenesulphonic acid was obtained in 90% yield by sulphonating and nitrating chlorobenzene and then reducing. C.O.C.

Influence of Non-planarity in Styrene and Stilbene Derivatives. III—Syntheses and Ultraviolet Spectra of 4,4′-substituted *Cis*- and *Trans*-Stilbenes

G. Riezebos and E. Havinga

Rec. Trav. chim., **80** (May 1961) 446–467

The synthesis and ultraviolet absorption spectra are reported for a series of *cis*- and *trans*-stilbenes (I; R^1 = H, CN, or NO_2 and R^2 = H, CH_3, or OCH_3). The differences between the spectra of the *trans* cpd. and those of the sterically hindered *cis*-isomers and also the influence of the above substituents are discussed by means of a semiquantitative treatment, and the influence of the steric inhibition on coplanarity is deduced qualitatively.



(I)

H.H.H.

Photo-Ionisation of Triarylmethyl Leuconitriles

A. H. Sporer

Trans. Faraday Soc., **57** (June 1961) 983–991

Two photo-reactions of triarylmethyl leuconitriles have been observed: dye formation and cleavage of substituents on amine nitrogens. Cleavage is most easily observed in solvents of low dielectric constant. It also occurs in solvents of high dielectric constants but is masked by dye formation, which occurs with a much higher quantum yield. It is suggested that dye formation occurs when the molecule undergoes internal conversion. W.R.M.

Fluorine-containing Cyanine Dyes. IX—Cyanine Dyes from Derivatives of 5,6-(Difluoromethylene)-dihydroxybenzothiazole

L. M. Yagupol'skiĭ and V. I. Troitskaya

Zhur. obshch. khim., **31** (Feb 1961) 628–632

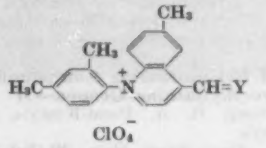
Synthesis of 2-methyl-5,6-difluoromethylene-dihydroxybenzothiazole and of 6-thiacarbocyanine dyes containing the difluoromethylenedihydroxy group (I) as substituent is described. It is shown that the introduction of I into the molecule of thiacyanine dyes causes a bathochromic shift (11–17 mμ) of the absorption maximum. G.J.K.

Synthetic Dyes. XX—Cyclisation of *p,p′*-Ditolylamine to Quaternary Lepidinium Salts and some of their Transformations

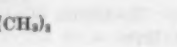
G. T. Pilyugin and B. M. Gutsulyak

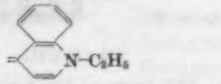
Zhur. obshch. khim., **31** (Feb 1961) 623–626

Quaternary *N*-*p*-tolyl-6-methyl-lepidinium perchlorate and picrate have been prepared. From the perchlorate 3 new dyes have been synthesised and their absorption spectra in ethanol measured. The dyes are

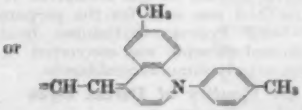


where Y is





or



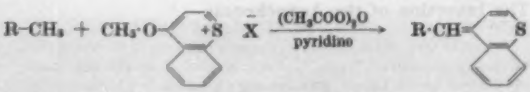
G.J.K.

Condensation of Tertiary Oxonium Chromone and Thiochromone Salts with Compounds containing Active Methyl or Methylene Groups

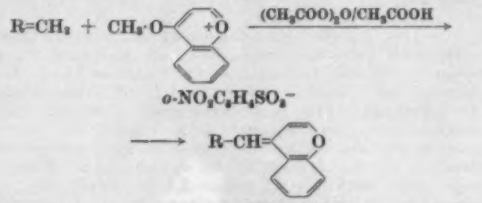
A. I. Kipryanov and A. I. Tolmachev

Zhur. obshch. khim., **30** (Feb 1960) 638–649

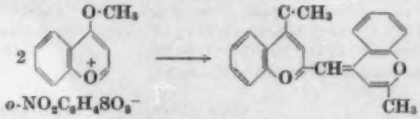
Tertiary oxonium salts of thiochromones condense with thiazoles with the formation of intensely coloured monomethinecyanine dyes. The reaction takes place according to the scheme



where R is 2-methyl-, 2-methyl-6-nitro-, or 2-methyl-α-naphtho-benzothiazole. The chromone oxonium salts react with methylene derivatives of thiazoles obtained by their treatment with caustic soda.

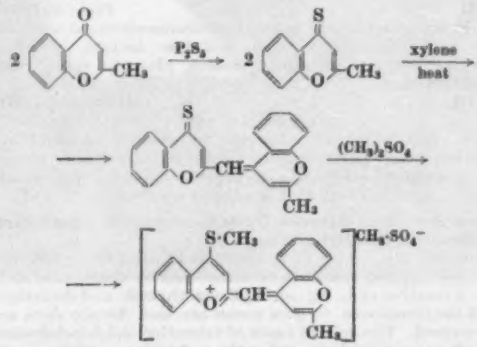


where R is 3-methyl(or ethyl)-benzothiazole, 3-ethyl-6-nitro-, or 3-methyl(or ethyl)-α-naphthothiazole. The oxonium salts of flavone and 5,6-benzochromone were similarly used. The monocyanines so produced are yellow and orange dyes. A monocyanine dye is prepared by the autocondensation of 2-methylchromone. Reaction takes place by heating on a water bath the chromone with *o*-$NO_2C_6H_4SO_3CH_3$ in toluene.

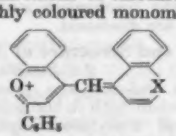


P_2S_5 converts 2-methylchromone to the thio derivative, which undergoes similar autocondensation.

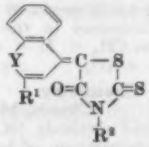
The compound so formed is converted into the dye with $(CH_3)_2SO_4$.



The condensation of 4-methylflavyl perchlorate with tertiary oxonium chromone or thiochromone leads to the formation of a highly coloured monomethinecyanine



where X is O or S. The tertiary thiochromone or flavone salts combine with heterocyclic systems containing an active methylene group to give cyanine dyes of the structure



where Y is S or O; R^1 is H or C_2H_5 and R^2 is CH_3 or C_6H_5.

T.Z.W.

Neutrocyanines

O. Riester *Chimia*, **15** (Jan 1961) 75–79

The structure and absorption maxima of neutrocyanines are discussed. The accepted formula, with charge distribution on the hetero atom, does not correspond to all the resonance possibilities in the molecule. A.T.P.

Hydrophilic Dye Cyanides. II—Rhodamine B (C.I. 45170) Cyanide

L. Chalkley *J. Org. Chem.*, **26** (1961) 408–412
Chem. Abs., **55** (10 July 1961) 13854

Rhodamine B cyanide (I) has a remarkably reactive nitrile group, in strong contrast to the behaviour of the familiar triphenylmethane dye cyanides. In aqueous solution it rapidly reverts at room temperature to the parent dye (II) and NaCN. In dil. mineral acid the nitrile group hydrolyses at room temperature to give 3,6-bis-(diethylamino) - 9 - carboxy - 9 - (2 - carboxyphenyl)-xanthene (III). Photolysis of I may yield not only II but also cornflower blue (IV). III is both photosensitive and a strong reducing agent, being readily oxidised to CO_2 and II. IV is relatively stable to acid but is destroyed by alkali. C.O.C.

Photoreduction of Neutral Red (C.I. 50040)

R. Havemann, H. Pietsch, and D. Ollmann
Z. physik. Chem. (Leipzig), **215** (1960) 388
Chem. Abs., **55** (10 July 1961) 13853

Depending on the pH of the solution different reduction products are obtained. At pH 0·5 photochemical bleaching is possible in presence of $SnCl_2$, leading to a yellow compound. At pH — 0·05 reduction with bleaching occurs in the dark, leading to a colourless compound. C.O.C.

Determination of Proton Equilibria for Neutral Red (C.I. 50040), Phenosafranine (C.I. 50200) and Safranine T (C.I. 50240)

T. R. Havemann, H. Pietsch, and D. Ollmann
Z. wiss. Phot., **54** (1960) 198–207
Chem. Abs., **55** (10 July 1961) 13141

Spectrophotometric measurements of the *pKs* of the above dyes, by determining the dependence of the extraction on pH, indicated that in all three dyes the free electron pair of the N atoms made possible the proton binding. C.O.C.

Reasons for the Formation and Nature of Impurities in Brilliant Green C (C.I. 59825)

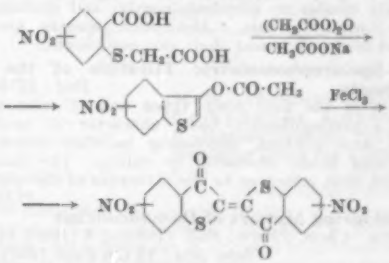
P. P. Karpukhin and A. G. Belobrov
Trudy Khar'kov. Politekh. Inst. im. V.I. Lenina, **26**, *Ser. Khim.-Tekhnol.*, (6) (1959) 93–100
Chem. Abs., **55** (26 June 1961) 12857

This dye is obtained by oxidising violanthrone with MnO_2 to the 16,17-quinone, which is then converted to 16,17-dihydroxyviolanthrone and methylated. The final product contains by-products which render it duller. Chromatographic analysis indicated that oxidation does not stop at the quinone, but goes further to yield some trioxy- and tetroxy-derivatives, 15,16,17-trimethoxy-violanthrone and 15,16,17,18-tetramethoxyviolanthrone having been found in the dye. Oxidising violanthrone with MnO_2 or MnO_2 yields a purer product (less further oxidation) than with $K_2Cr_2O_7$. MnO_2 in strong H_2SO_4 is the best, and addition of H_3BO_3 is beneficial. During methylation the first methyl group enters without difficulty, but introduction of the second group meets with steric hindrance. In C.I. 59825, 16,17-dimethoxyviolanthrone, the vicinal methoxy groups are under strain, and although the planarity is not destroyed, the valence angles are affected. This is shown by the absorption spectrum of this dye exhibiting lower intensity than that of violanthrone and also a strong bathochromic shift (43 mμ). However, at 200°C the maximum absorption moves to shorter wavelengths and the intensity increases. The planarity of the dye is affected at high temperatures. Hence a high temperature is needed for complete methylation. If methylation is done at a low temperature, a certain proportion of an intermediate monomethylated product is formed. In addition to these by-products caused by over-oxidation and under-methylation, another impurity was found which could not be identified. C.O.C.

Influence of Substitution on Thioindigoid Dyes. III—Dinitrosubstituted Thioindigo

N. S. Dokunikhin and Yu. E. Gerasimenko
Zhur. obshch. khim., **30** (Feb 1960) 635–638

Three symmetrical dinitrothioindigoid dyes were prepared according to the scheme—



The nitro groups were in 5,5′-, 6,6′-, and 7,7′-positions respectively. The yield of the dye was improved by use of bromine as catalyst. It was found that an NO_2 group *o*- or *p*- to the S atom causes a hypsochromic shift of the absorption spectrum but *meta*-substitution gives a bathochromic shift. When an electron-donating substituent is present *o*- or *p*- to the S atom the colour of the dye is deepened; when in the *m*-position, the colour is lightened. T.Z.W.

Structure of Phthalocyanine (C.I. 74000)

N. A. Frigerio
J. Org. Chem., **26** (June 1961) 2115–2116

Of two possible structures for phthalocyanine—$(C_8H_4N_2)_4$ and $(C_8H_4N_2)_4H_2$—evidence for the latter was obtained during work on the syntheses of deuteriophthalocyanine. It has been found that 1/9th of the total hydrogen content of phthalocyanine is easily replaced by deuterium under conditions which result in no hydrogen–deuterium exchange with copper phthalocyanine. I.G.L.

Ultraviolet Spectra of *N*-Heterocyclic Systems. II—Spectra of 5-8-Hydroxyisoquinolines and Related Compounds

K. Nakanishi, M. Ohashi, S. Kumasaki, and H. Koike
Bull. Chem. Soc. Japan, **34** (Apr 1961) 533–537 (*in English*)

This study of the spectra of hydroxyisoquinolines (in which the hydroxyl group is neither α nor γ to the ring nitrogen atom) was undertaken in connection with the study of natural products, e.g. the mould pigment monascorubin, shown to be a derivative of 5-, 6-, 7- or 8-hydroxyisoquinoline. The solvents employed were CH_3OH, 0·1-N HCl–CH_3OH and 0·1-N NaOH–CH_3OH, and the compounds investigated were 5-, 6-, 7- and 8-hydroxyisoquinolines, the methiodides of the 5-, 7- and 8-derivatives, and 6- and 7-methoxyisoquinolines. With the exception of the 6-hydroxy- and 6-methoxy-isoquinolines, the above derivatives, in common with other hydroxyazanaphthalenes, exhibit three major absorption peaks, and shifts in bands accompanying changes in media and conversion to derivatives closely parallel those observed in the case of 3-hydroxypyridine. Ionisation of the phenolic group causes a bathochromic shift, as does *N*-protonation and, to a greater extent, the change $N \rightarrow N^+CH_3$ (*N*-quaternisation). Replacement of –OH by $-OCH_3$ results in a slight hypsochromic shift. From these observations it proved possible to classify these hydroxy-azanaphthalenes as α- or β-naphthol types (after Ewing and Steck, *J. Amer. Chem. Soc.*, **68** (1946) 2181). 6-Hydroxy- and 6-methoxy-isoquinolines cannot, however, be classified in this way, and reasons for this are discussed. I.G.L.

Chemistry of the Bile Pigments

C. H. Gray, A. Lichtarowicz-Kulczycka, D. C. Nicholson, and (in part of II only) Z. Potryka

II—Preparation and Spectral Properties of Biliverdin

J.C.S., (June 1961) 2264–2268

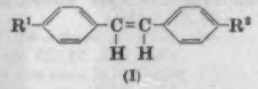
Newly determined absorption spectra between 250 and 700 mμ of specially purified biliverdin (I), reported for CH_3OH, $CHCl_3$, and methanolic HCl soln., show a difference from the data extant, where the preparations used

Influence of Non-planarity in Styrene and Stilbene Derivatives. III—Syntheses and Ultraviolet Spectra of 4,4′-substituted *Cis*- and *Trans*-Stilbenes

G. Riezebos and E. Havinga

Rec. Trav. chim., **80** (May 1961) 446–467

The synthesis and ultraviolet absorption spectra are reported for a series of *cis*- and *trans*-stilbenes (I; R^1 = H, CN, or NO_2 and R^2 = H, CH_3, or OCH_3). The differences between the spectra of the *trans* cpd. and those of the sterically hindered *cis*-isomers and also the influence of the above substituents are discussed by means of a semiquantitative treatment, and the influence of the steric inhibition on coplanarity is deduced qualitatively.



(I)

H.H.H.

Photo-ionisation of Triarylmethyl Leuconitriles

A. H. Sporer

Trans. Faraday Soc., **57** (June 1961) 983–991

Two photo-reactions of triarylmethyl leuconitriles have been observed: dye formation and cleavage of substituents on amine nitrogens. Cleavage is most easily observed in solvents of low dielectric constant. It also occurs in solvents of high dielectric constants but is masked by dye formation, which occurs with a much higher quantum yield. It is suggested that dye formation occurs when the molecule undergoes internal conversion. W.R.M.

Fluorine-containing Cyanine Dyes. IX—Cyanine Dyes from Derivatives of 5,6-(Difluoromethylene)-dihydroxybenzothiazole

L. M. Yagupol'skiĭ and V. I. Troitskaya

Zhur. obshch. khim., **31** (Feb 1961) 628–632

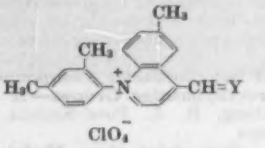
Synthesis of 2-methyl-5,6-difluoromethylene-dihydroxybenzothiazole and of 6-thiacarbocyanine dyes containing the difluoromethylenedihydroxy group (I) as substituent is described. It is shown that the introduction of I into the molecule of thiacyanine dyes causes a bathochromic shift (11–17 mμ) of the absorption maximum. G.J.K.

Synthetic Dyes. XX—Cyclisation of *p,p′*-Ditolylamine to Quaternary Lepidinium Salts and some of their Transformations

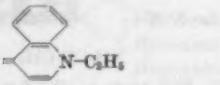
G. T. Pilyugin and B. M. Gutsulyak

Zhur. obshch. khim., **31** (Feb 1961) 623–626

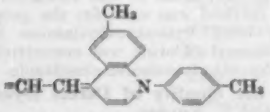
Quaternary *N*-*p*-tolyl-6-methyl-lepidinium perchlorate and picrate have been prepared. From the perchlorate 3 new dyes have been synthesised and their absorption spectra in ethanol measured. The dyes are



where Y is



or



G.J.K.

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A. I. Kipryanov and A. I. Tolmachev

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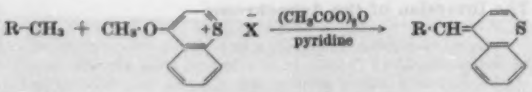
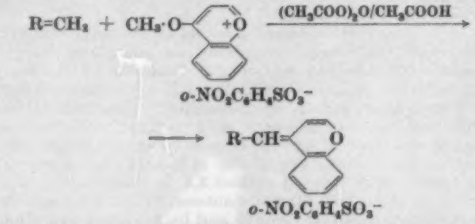
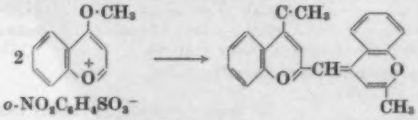


where R is 2-methyl-, 2-methyl-6-nitro-, or 2-methyl-α-naphtho-benzothiazole. The chromone oxonium salts react with methylene derivatives of thiazoles obtained by their treatment with caustic soda.

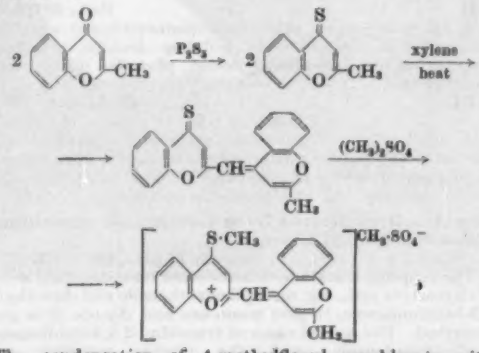


where R is 3-methyl(or ethyl)-benzothiazole, 3-ethyl-6-nitro-, or 3-methyl(or ethyl)-α-naphthothiazole. The oxonium salts of flavone and 5,6-benzochromone were similarly used. The monocyanines so produced are yellow and orange dyes. A monocyanine dye is prepared by the autocondensation of 2-methylchromone. Reaction takes place by heating on a water bath the chromone with *o*-$NO_2C_6H_4SO_3CH_3$ in toluene.



P_2S_5 converts 2-methylchromone to the thio derivative, which undergoes similar autocondensation.

The compound so formed is converted into the dye with $(CH_3)_2SO_4$.



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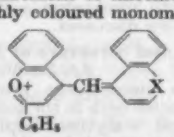
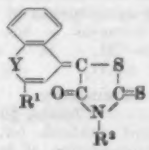


where X is O or S. The tertiary thiochromone or flavone salts combine with heterocyclic systems containing an active methylene group to give cyanine dyes of the structure



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T.Z.W.

Neutrocyanines
O. Riester *Chimia*, **15** (Jan 1961) 75–79

The structure and absorption maxima of neutrocyanines are discussed. The accepted formula, with charge distribution on the hetero atom, does not correspond to all the resonance possibilities in the molecule. A.T.P.

Hydrophilic Dye Cyanides. II—Rhodamine B (C.I. 45170) Cyanide
L. Chalkley *J. Org. Chem.*, **26** (1961) 408–412
Chem. Abs., **55** (10 July 1961) 13854

Rhodamine B cyanide (I) has a remarkably reactive nitrile group, in strong contrast to the behaviour of the familiar triphenylmethane dye cyanides. In aqueous solution it rapidly reverts at room temperature to the parent dye (II) and NaCN. In dil. mineral acid the nitrile group hydrolyses at room temperature to give 3,6-bis-(diethylamino) - 9 - carboxy - 9 - (2 - carboxyphenyl)-xanthene (III). Photolysis of I may yield not only II but also cornflower blue (IV). III is both photosensitive and a strong reducing agent, being readily oxidised to CO_2 and II. IV is relatively stable to acid but is destroyed by alkali. C.O.C.

Photoreduction of Neutral Red (C.I. 50040)
R. Havemann, H. Pietsch, and D. Ollmann
Z. physik. Chem. (Leipzig), **215** (1960) 388
Chem. Abs., **55** (10 July 1961) 13853

Depending on the pH of the solution different reduction products are obtained. At pH 0·5 photochemical bleaching is possible in presence of $SnCl_2$, leading to a yellow compound. At pH −0·05 reduction with bleaching occurs in the dark, leading to a colourless compound. C.O.C.

Determination of Proton Equilibria for Neutral Red (C.I. 50040), Phenosafranine (C.I. 50200) and Safranine T (C.I. 50240)
T. R. Havemann, H. Pietsch, and D. Ollmann
Z. wiss. Phot., **54** (1960) 198–207
Chem. Abs., **55** (10 July 1961) 13141

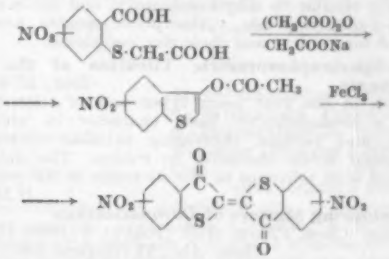
Spectrophotometric measurements of the *pKs* of the above dyes, by determining the dependence of the extraction on pH, indicated that in all three dyes the free electron pair of the N atoms made possible the proton binding. C.O.C.

Reasons for the Formation and Nature of Impurities in Brilliant Green C (C.I. 59825)
P. P. Karpukhin and A. G. Belobrov
Trudy Khar'kov. Politekh. Inst. im. V.I. Lenina, **26**, *Ser. Khim.-Tekhnol.*, (6) (1959) 93–100
Chem. Abs., **55** (26 June 1961) 12857

This dye is obtained by oxidising violanthrone with MnO_2 to the 16,17-quinone, which is then converted to 16,17-dihydroxyviolanthrone and methylated. The final product contains by-products which render it duller. Chromatographic analysis indicated that oxidation does not stop at the quinone, but goes further to yield some trioxy- and tetroxy-derivatives, 15,16,17-trimethoxy-violanthrone and 15,16,17,18-tetramethoxyviolanthrone having been found in the dye. Oxidising violanthrone with MnO_2 or MnO_2 yields a purer product (less further oxidation) than with $K_2Cr_2O_7$. MnO_2 in strong H_2SO_4 is the best, and addition of H_2BO_3 is beneficial. During methylation the first methyl group enters without difficulty, but introduction of the second group meets with steric hindrance. In C.I. 59825, 16,17-dimethoxyviolanthrone, the vicinal methoxy groups are under strain, and although the planarity is not destroyed, the valence angles are affected. This is shown by the absorption spectrum of this dye exhibiting lower intensity than that of violanthrone and also a strong bathochromic shift (43 mμ). However, at 200°C the maximum absorption moves to shorter wavelengths and the intensity increases. The planarity of the dye is affected at high temperatures. Hence a high temperature is needed for complete methylation. If methylation is done at a low temperature, a certain proportion of an intermediate monomethylated product is formed. In addition to these by-products caused by over-oxidation and under-methylation, another impurity was found which could not be identified. C.O.C.

Influence of Substitution on Thioindigoid Dyes. III—Dinitrosubstituted Thioindigo
N. S. Dokunikhin and Yu. E. Gerasimenko
Zhur. obshch. khim., **30** (Feb 1960) 635–638

Three symmetrical dinitrothioindigoid dyes were prepared according to the scheme—



The nitro groups were in 5,5′-, 6,6′-, and 7,7′-positions respectively. The yield of the dye was improved by use of bromine as catalyst. It was found that an NO_2 group *o*- or *p*- to the S atom causes a hypsochromic shift of the absorption spectrum but *meta*-substitution gives a bathochromic shift. When an electron-donating substituent is present *o*- or *p*- to the S atom the colour of the dye is deepened; when in the *m*-position, the colour is lightened. T.Z.W.

Structure of Phthalocyanine (C.I. 74000)
N. A. Frigerio
J. Org. Chem., **26** (June 1961) 2115–2116

Of two possible structures for phthalocyanine—$(C_8H_4N_2)_4$ and $(C_8H_4N_2)_4H_2$—evidence for the latter was obtained during work on the syntheses of deuteriophthalocyanine. It has been found that 1/9th of the total hydrogen content of phthalocyanine is easily replaced by deuterium under conditions which result in no hydrogen–deuterium exchange with copper phthalocyanine. I.G.L.

Ultraviolet Spectra of *N*-Heterocyclic Systems. II—Spectra of 5-8-Hydroxyisoquinolines and Related Compounds
K. Nakanishi, M. Ohashi, S. Kumasaki, and H. Koike
Bull. Chem. Soc. Japan, **34** (Apr 1961) 533–537 (*in English*)

This study of the spectra of hydroxyisoquinolines (in which the hydroxyl group is neither α nor γ to the ring nitrogen atom) was undertaken in connection with the study of natural products, e.g. the mould pigment monascorubin, shown to be a derivative of 5-, 6-, 7- or 8-hydroxyisoquinoline. The solvents employed were CH_3OH, 0·1-N HCl–CH_3OH and 0·1-N NaOH–CH_3OH, and the compounds investigated were 5-, 6-, 7- and 8-hydroxyisoquinolines, the methiodides of the 5-, 7- and 8-derivatives, and 6- and 7-methoxyisoquinolines. With the exception of the 6-hydroxy- and 6-methoxy-isoquinolines, the above derivatives, in common with other hydroxyazanaphthalenes, exhibit three major absorption peaks, and shifts in bands accompanying changes in media and conversion to derivatives closely parallel those observed in the case of 3-hydroxypyridine. Ionisation of the phenolic group causes a bathochromic shift, as does *N*-protonation and, to a greater extent, the change $N \rightarrow N^+CH_3$ (*N*-quaternisation). Replacement of –OH by $-OCH_3$ results in a slight hypsochromic shift. From these observations it proved possible to classify these hydroxy-azanaphthalenes as α- or β-naphthol types (after Ewing and Steck, *J. Amer. Chem. Soc.*, **68** (1946) 2181). 6-Hydroxy- and 6-methoxy-isoquinolines cannot, however, be classified in this way, and reasons for this are discussed. I.G.L.

Chemistry of the Bile Pigments
C. H. Gray, A. Lichtarowicz-Kulczycka, D. C. Nicholson, and (in part of II only) Z. Potryka

II—Preparation and Spectral Properties of Biliverdin *J.C.S.*, (June 1961) 2264–2268

Newly determined absorption spectra between 250 and 700 mμ of specially purified biliverdin (I), reported for CH_3OH, $CHCl_3$, and methanolic HCl soln., show a difference from the data extant, where the preparations used

were probably contaminated with violenoid pigments and residual bilirubin.

III—Prototropy of Bilirubin to a Verdinoid Pigment *Ibid.*, 2268–2275

Treatment of I with alkoxides in absence of oxygen produces, by prototropic change, a pigment spectroscopically similar to dihydrobiliverdin and containing a β-ethyl-β-methylpyrrolo. Absorption spectra are also reported for verdins and their zinc complexes.

IV—Spectrophotometric Titration of the Bile Pigments *Ibid.*, 2276–2283

Curves of the four main types and of related cpd. reveal a predominantly basic character in urobilins, violins, and verdins, decreasing in that order, and pronounced acidic character in rubins. The data are discussed with reference to the structure of the pigment. H.H.H.

The Colouring Matters of *Damnacanthus*

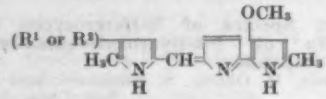
Y. Hirose *Chem. Pharm. Bull.* (Tokyo), **8** (1960) 417–426
Chem. Abs., **55** (26 June 1961) 12372

Evidence is presented to show that damnacanthal is the 1,2,3-$CH_3O(CHO)(OH)$ derivative of anthraquinone, damnacanthol the 1,2,3-$CH_3O(OHCH_2)(OH)$, norjuzunal the 2,1,3,5-$OHC(HO)_3$, norjuzunol the 2,1,3,5-$HOCH_2(HO)_3$, juzunal the 2,1,3,5-$OHC(CH_3O)(HO)_2$ and juzunol the 2,1,3,5-$HOCH_2(CH_3O)(HO)_2$ derivative. They are the first naturally occurring 1,3,5-trihydroxyanthraquinones to be discovered. C.O.C.

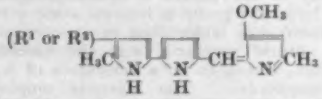
Structure of the Red Pigment, Prodigiosine, and a Pigment resembling it

G. Narni and R. A. Nicolaus
Rend. accad. sci. fis. e mat., (*Soc. nazl. sci., Napoli*), **26** (1959) 7 pp.
Chem. Abs., **55** (15 May 1961) 9370

Prodigiosine, a red pigment, was obtained from *Serratia marcescens* and on the basis of its chemical properties and the close relationship existing between prodigiosine (R^1 = amyl) and a prodigiosine-like pigment ($R^2 = C_9H_{19}$) two possible structures are suggested



and



C.O.C.

Linearly Conjugated Polyene Systems. I—Comparative Spectroscopy of all-*trans* Carotenoids

J. Szoke *Magyar Tudományos Akad. Kém. Tudományok Osztályának, Közleményei*, **14** (1960) 11–22
Chem. Abs., **55** (29 May 1961) 10068

Absorption maxima of carotenoids, hydroxycarotenoids, epoxycarotenoids, and furanocarotenoids were determined in CS_2 to find the effects of the functional groups within the molecules on the absorption spectra. An isolated double bond has no effect, but β-type double bonds have an effect, about 20% of that of chain double bonds, on absorption maxima. One or two OH groups in the 3-position have no effect (Karrer and Jucker [*Carotenoids* (1950); *Chem. Abs.*, **45** (1951) 3892] state that OH groups in 3-position exert weak (1–2 mμ) hypsochromic effect). An OH group α- to the conjugated system (or further away) has no effect, nor has presence of one or two epoxy groups. A furanoid group has no effect. C.O.C.

Pigments from the Ferric Sediment obtained during the Reduction of Nitrobenzene

I. Reichel, I. Philips, and E. Secosan
Bul. ştiint. tehn., Inst. politehn. Timişoara (2) **1** (**15**) (1956) 361–373
Chem. Abs., **55** (12 June 1961) 11876

The following conditions are necessary to produce the best-quality pigments: avoid excessive alkalisation of the reduction mixture, prevent formation of the addition compound $FeCl_2 \cdot 2C_6H_5NH_2$ by adding a little Fe powder at the end of the reduction, and avoid disintegration of the end-products. It is best to recover red and black but not yellow pigments. C.O.C.

Dihydroxyacetone as a Skin-colouring Agent

L. Goldman, J. Barkoff, D. Blaney, T. Nakai, and R. Suskind
J. Amer. Acad. Gen. Pract., **22** (3) (1960) 96–98
Chem. Abs., **55** (26 June 1961) 12777

$HOCH_2COCH_2OH$, hitherto believed to be non-injurious when used in cosmetics, causes eczematous dermatitis when used as a skin colorant in cases of vitiligo and other blemishes. It is useless as a hair dye. C.O.C.

Synthesis of Yellow and Red "Cadmapone" Pigments and Study of their Physicotechnical Properties

N. S. Rassudova, N. A. Tikhonova, and A. A. Butkute
Lakokrasochnye Materialy i ikh Primenenie, (1960) (5) 22–24
Chem. Abs., **55** (26 June 1961) 12881

13–15% aq. BaS or BaS + BaSe was added to warm 28–30% aq. $CdSO_4$ and the precipitate heated in absence of air. The best conditions for matching the colour of conventional CdS(Se) pigments (cf. C.I. 77117 and 77202) (for yellow first, then for orange–red) were: mix at 50–55°C, 60–70°C; heat precipitate at 650–670°C, 530–550°C; duration of heating 40 min, 45–60 min. Pigments obtained in this manner have better covering power than conventional CdS(Se) pigments or mechanical mixtures of CdS(Se) and $BaSO_4$ because of their high sp. surface area and content of smaller particles ($\leqslant 0{\cdot}75\,\mu$). The yellow is more brilliant than the conventional pigment or mechanical mixtures, but the red is less brilliant. Pigments made in this way are unaffected by 350 h exposure in a Weather-Ometer. For CdS : CdSe ratios of from 7·33 to 0·25 the hue deepens from orange to dark cherry. The oil capacities and spread areas of various types of pigments produced in this manner are given. X-ray analysis showed them to be mixtures of CdS (or CdS and CdSe) with $BaSO_4$. C.O.C.

Production of Red Lead (C.I. 77578)

N. V. Ogarev *Trudy Leningrad. Tekhnol. Inst. im. Lensoveta*, (50) (1959) 274–279
Chem. Abs., **55** (10 July 1961) 13783

Oxidation of PbO in a fluidised layer produces Red Lead 7–8 times more quickly than the conventional method. The operational life of the reactor is considerably lengthened by use of ceramic parts. Consumption of fuel is decreased and the apparatus can be enclosed, thus preventing atmospheric pollution by Pb oxides. C.O.C.

Increase in the Chalking Resistance of Zinc Whites (C.I. Pigment White 4) under Atmospheric Conditions

N. K. Piktorinskaya, D. M. Shub, M. L. Borodina, and P. M. Bogatyrev
Lakokrasochnye Materialy i ikh Primenenie, (6) (1960) 21–26
Chem. Abs., **55** (10 July 1961) 13869

The small needle-shaped Zinc Whites obtained by the muffle method from pure Zn in absence of producer gas show most chalking. Introduction of purified producer gas into the muffle furnace gives the pigments greater stability. Presence of small amounts of Cu, Cd, and Pb in the metallic Zn also increases the stability of the resultant pigments. The photoelectrochemical method rapidly determines the atmospheric stability of ZnO and can completely replace Weather-Ometer tests. Presence of producer gas somewhat increases the soluble SO_4^{2-} content in ZnO but this can be corrected by addition of 0·5% of $BaCO_3$. C.O.C.

Fundamental Processes in Fluorescence Phenomena

A. Kling and J. Kurz
Melliand Textilber., **42** (June 1961) 698–701

The internal processes which produce fluorescence are discussed and the various methods for extinguishing it are described. The importance of the information recorded for the textile processer is indicated. H.H.H.

PATENTS

Colour Couplers

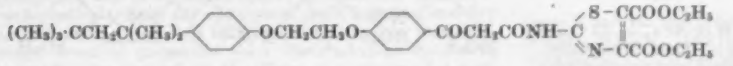
Gevaert Photo-Producten *BP* 873,124 (27 July 1956)

A silver halide image is developed with a primary aromatic amino developer in presence of Na 5-(*p*-cetylbenzoylacetylamino)-2-methylbenzenesulphonate, Na

o-(*p*-cetylbenzoylacetylamino) benzenesulphonate, *N*-(*p*-cetylbenzoylacetyl)-5-aminoisophthalic acid, or Na *p*-[*p*-(*p*-cetyloxybenzoylacetylamino)-phenylsulphonylamino]-benzene sulphonate. The resulting dye images are fast to diffusion and other agencies and have particularly favourable absorption characteristics.

BP 873,125 (19 June 1957)

Images of increased colour density are obtained by using an aromatic amino developer and a benzoyl acetamide colour coupler whose benzoyl group contains an alkoxy substituent containing a group conferring fastness to diffusion and whose amino group is substituted by a heterocyclic nucleus, e.g.



yields a yellow image. C.O.C.

Monoazo Disperse Dyes from *NN*-Bis(cyanoethyl)-anilines

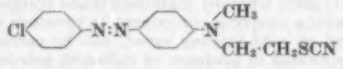
Ciba *BP* 872,481 (Switzerland 11 Oct 1957)

Diazotised benzene-series amines containing Alk, Hal, alkylsulphonyl, sulphamyl, etc., substituents but no SO_3H or COOH groups, are diazotised and coupled with *NN*-bis(cyanoethyl)anilines which may contain Hal, OAlk, Alk, etc., groups in the 2- and/or 5-positions to give yellow, orange, and red disperse dyes. Thus diazotised 2-cyano-4-nitroaniline coupled with *NN*-bis(cyanoethyl)-aniline gives a red. E.S.

Monoazo Disperse Dyes containing Thiocyanoalkyl Groups

FBy *BP* 870,580 (Germany 25 Nov 1957)

Diazo compounds of the benzene, naphthalene, thiazole, or thiodiazole series free from solubilising groups are coupled with benzene-series amines containing *N*-thiocyanoalkyl groups to give disperse dyes. Thus *p*-chloroaniline diazotised and coupled with *N*-methyl-*N*-β-thiocyanatoaniline gives

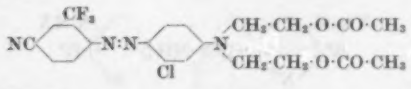


which dyes cellulose acetate, polyamide, and polyester fibres yellow. E.S.

Monoazo Disperse Dyes

ICI *BP* 872,204 (23 Dec 1958)

Orange to scarlet disperse dyes are prepared by coupling diazotised 2-amino-5-cyanobenzotrifluoride with *NN*-bis(β-acyloxyethyl)aniline, which may carry CH_3Cl, etc. in the *m*-position; the acyl group has < 6 C. Or acylation of the OH groups may follow coupling. Thus the monoazo compound

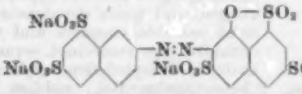


is an orange. E.S.

Azonaphthalene Dyes containing Three or Four Sulpho Groups

Eastman Kodak Co. *USP* 2,956,879 (7 Jan 1958)

Dyes of formula $R^1{\cdot}N{:}N{\cdot}R^2$ (R^1 and R^2 = same or different naphthalene groups each containing at least one sulpho substituent and together 3 or 4 sulpho groups), e.g.

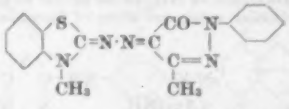


are excellent filter and absorbing dyes for use in silver halide emulsions sensitised to the green region of the spectrum. They have no sensitising or desensitising properties. C.O.C.

Basic Monoazo Dyes

BASF *BP* 871,426 (Germany 9 Sept 1958)

By condensing azo coupling components in presence of oxidising agents with sulphonyl derivatives of the hydrazones used in *BP* 782,310 (*J.S.D.C.*, **73** (1957) 526), improved yield and smoothness of operation are obtained. Thus, 3-methylbenzothiazolone-(2)-benzenesulphonyl hydrazone and 3-methyl-1-phenyl-5-pyrazolone dissolved in *N*-methylpyrrolidone containing aq. NaOH are treated with potassium ferricyanide to give the red compound

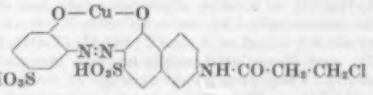


E.S.

Metal-complex Monoazo Compounds Containing β-Haloacylamide Groups—Reactive Dyes for Cellulose

Ciba *BP* 870,047 (Switzerland 5 Mar 1957)

Metal-complexes of *oo'*-dihydroxy- or *oo'*-carboxy-hydroxy-monoazo compounds containing a primary or secondary amino group are *N*-acylated with the anhydride or halide of a β-halogenated fatty acid, e.g. butyric or ethanesulphonic acid, to give the title dyes. Thus the alkali-coupled monoazo compound 2-aminophenol-4-sulphonic acid → J acid is converted into the Cu-complex, which is then acylated with β-chloropropionyl chloride to give

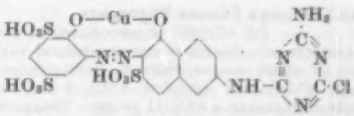


which, padded on cotton and steamed in presence of NaOH, gives a red. E.S.

Metal-complex Monoazo Reactive Dyes for Cellulose

Ciba *BP* 870,985 (Switzerland 10 Aug 1956)

The title dyes are the copper, and the 1:2 chromium and cobalt, complexes of *oo'*-dihydroxymonoazo compounds containing > 1 (and preferably > 2) SO_3H groups, and a triazine ring containing 1 Hal and 1 amino or substituted amino group. Thus diazotised 2-aminophenol-4,6-disulphonic acid is alkali-coupled with J acid, and the product is heated with aq. $CuSO_4$ to give the metal complex. Condensation at the NH_2 group with 1 mol. of cyanuric chloride is then effected, and treatment with ammonia then replaces the second Cl atom in the triazine ring, giving

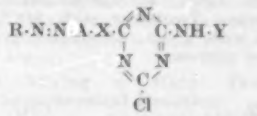


which gives ruby red on cotton when applied in presence of aq. NaOH. E.S.

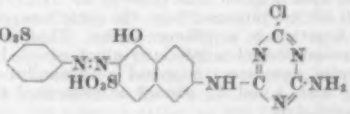
Monoazo Reactive Dyes for Cellulose

Ciba *BP* 872,249 (Switzerland 14 Sept 1956)

The title dyes have formula



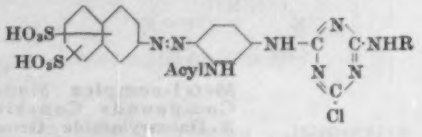
(R = residue of diazo component preferably containing COOH or SO_3H groups; A = naphthalene residue having OH *ortho* to the azo link; X = –NH– or –NAlk– and is in a β-position in A; Y = H or radical of < 13 C). Thus the alkali-coupled monoazo compound metanilic acid → J acid is condensed with 1 mol. of cyanuric chloride, and a second Cl atom in the triazine ring is then replaced by stirring with ammonia, giving the orange reactive dye



E.S.

Mono- and Dis-azo Reactive Dyes for Cellulose
Ciba *BP* 869,279 (Switzerland 14 Sept 1956)

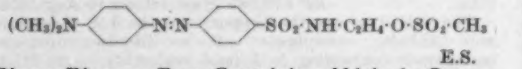
Aminomono- and aminodis-azo dyes containing at least 2 SO_3H and/or COOH groups are condensed with 1 mol. of cyanuric chloride, a second Cl atom of which is then condensed with a mol. of NH_3 or an amine to give the title dyes. Of particular value are dyes of formula



(the acyl group has up to 4 C; R = H or Ar of < 13 C and contains a COOH or SO_3H group). Thus 2-naphthylamine-4,8-disulphonic acid is diazotised and coupled with *m*-aminoacetanilide in presence of acetic acid, the product is condensed with 1 mol. of cyanuric chloride, and ammonia is then added to give the reddish yellow reactive dye. E.S.

Disazo Azo Dyes containing Sulphonic Ester Groups
ICI *BP* 869,100 (29 Oct 1958)

The title dyes are prepared by esterifying the OH groups in compounds $D(SO_3(NX)_{n-1}\cdot A\cdot OH)_m$ with an acid halide $-R\cdot SO_2\cdot Hal$ (D = residue of mono- or dis-azo dye; X = H, Alk, monocyclic Ar, or $-A\cdot O\cdot SO_2\cdot R$; R = Alk or monocyclic Ar; *n* and *m* = 1 or 2; SO_3H, COOH, $SO_2\cdot NH_2$, $SO_2\cdot Alk$, and *N*-acylated sulphonamido groups may not be present). They give good coverage of irregular-dyeing polyamide fibres. Thus the monoazo compound *p*-aminobenzenesulphon-*N*-β-hydroxyethylamide → *NN*-dimethylaniline is stirred in pyridine with methanesulphonyl chloride to give the yellowish orange



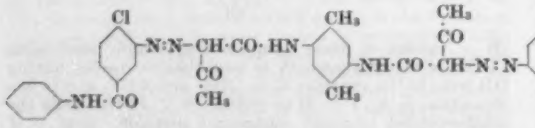
E.S.

Disazo Disperse Dyes Containing Aldehyde Groups
FBy *BP* 871,633 (Germany 1 Aug 1958)

The title dyes are of the type amino-benzo- or amino-naphtho-aldehyde → primary arylamine → phenol, and give yellow, orange, and brown of good sublimation fastness on polyester fibres. Thus the disazo compound *p*-aminobenzaldehyde → *m*-toluidine → phenol is a yellow. E.S.

Yellow and Orange Disazo Pigments
Ciba *BP* 873,003 (Switzerland 17 May 1957)

Aromatic primary diamines are condensed with 2 mol. of diketen or ethyl acetoacetate to give bis-acetoacetyl compounds which are coupled with 2 mol. of diazo components containing a COOH group. Treatment with, e.g., $SOCl_2$ converts the COOH groups into COCl, and condensation with primary arylamines, e.g. aniline, then converts these groups into arylamide groups. The products are greenish yellow to orange pigments of good fastness to light, heat, and migration into solvents. Thus 2 mol. of diazotised 3-amino-4-chlorobenzoic acid are coupled with 1 mol. of 2,5-bis-acetoacetylamino-1,4-dimethylbenzene. Treatment with $SOCl_2$, followed by condensation with 2 mol. of aniline, gives the bright reddish yellow pigment



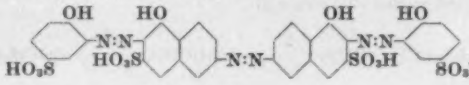
E.S.

Green Trisazo Leather Dyes
BASF *BP* 870,698 (Germany 19 Dec 1958)

Trisazo dyes brighter than those of *BP* 785,101 (J.S.D.C., **74** (1958) 59) are prepared from the same components but coupled together in a different order. Thus tetrazotised 4,4′-diaminodiphenyl sulphide is alkali-coupled with the acid-coupled monoazo compound *p*-nitroaniline→H acid. Coupling with 1 mol. of phenol or resorcinol then gives the required dye. E.S.

Copperable Trisazo Direct Dyes
FBy *BP* 872,685 (Germany 28 Feb 1958)

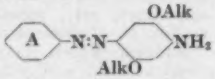
The title dyes are made by alkali-coupling 5,5′-dihydroxy-2,2′-azonaphthalene-7,7′-disulphonic acid (I) with 2 mol. of diazotised amines having chelatable (or potentially chelatable) groups in the *o*-position to the diazo group. Coppering may be effected on the fibre or in substance, and alternative syntheses of the same dyes are available. Thus diazotised *O*-benzenesulphonyl-J acid is treated with a sulphite to give I. Coupling with 2 mol. of diazotised 2-aminoanisole-4-sulphonic acid, and coppering with demethylation, gives the Cu-complex of



which dyes cotton bluish violet. E.S.

Azoic Blues for Cellulose Acetate and Polyester Fibres
FH *BP* 872,948 (Germany 19 Dec 1956)

Aminoazo compounds



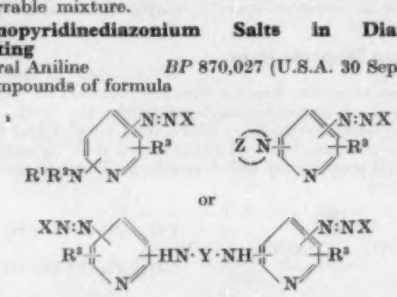
(the nucleus A may contain Alk, OAlk and/or Hal substituents) are diazotised and coupled on the fibre with the anilide, toluidides, or alkoxyanilides of 3-hydroxy-2-naphthoic acid to give blues. Thus the aminoazo compound *p*-chloroaniline → 2,5-dimethoxyaniline is applied to polyethyleneglycol terephthalate with 3-hydroxy-2-naphtho-*o*-anisidide at 120°C in presence of tetrahydronaphthalene as carrier. Treatment in hot aq. nitrous acid then gives a navy blue. E.S.

Preparation of Azo Pigments in Absence of Water
Ciba *BP* 872,416 (Switzerland 27 Mar 1957)

The process described in *BP* 861,443 (J.S.D.C., **77** (June 1961) 260), whereby stabilised diazo compounds are kneaded with a coupling component in presence of a little solvent and an acid-binding agent, can be carried out in simpler apparatus in presence of sufficient solvent to give a stirrable mixture. E.S.

Aminopyridinediazonium Salts in Diazotype Printing
General Aniline *BP* 870,027 (U.S.A. 30 Sept 1958)

Compounds of formula



(R^1 = H, Alk, Ar or aralkyl; R^2 = Alk, Ar or aralkyl; Z = atoms to complete a 6-membered hydrogenated ring; R^3 = H or Alk; X = anion; Y = diphenyl or benzylphenyl), e.g. 6-dimethylamino-3-pyridinediazonium chloride and its $ZnCl_2$, $SnCl_4$, $CdCl_2$, $HgCl_2$, and BF_3 double salts, when used in diazotype materials, give speed of exposure, dense images, and quick development. The most suitable couplers and the hues obtained are: resorcinol, sepia; 6,7-dihydroxy-2-naphthalenesulphonic acid, reddish purple; 2,3-naphthalenediol, reddish purple; acetoacetanilide, yellow; phloroglucinol, reddish brown; *m*-hydroxyphenylurea, sepia; *N*-benzylacetoacetamide, yellow; 3-methyl-1-phenyl-5-pyrazolone, orange; *N*-*o*-methoxyphenyl-3-hydroxy-2-naphthamide, bluish purple; 4,4′-thiodiresorcinol, red to sepia. C.O.C.

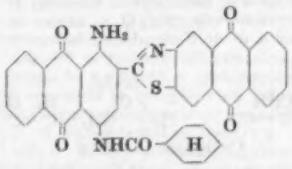
Anthraquinone Dyes for Polyolefins
Acna *BP* 872,882 (Italy 11 May 1959)

Anthraquinone dyes containing in at least one α-position an alkylamino group (Alk of 2–18 C) or a cycloalkylamino

group have good affinity for polyolefins. Thus, polypropylene is readily dyed a brilliant reddish blue of good fastness by 1,4-di(butylamino)anthraquinone from a soap bath in absence of any other assistant. If desired, dyeing may be carried out at > 100°C. C.O.C.

Anthraquinone Vat Dyes
Ciba *BP* 873,617 (Switzerland 12 Dec 1956)

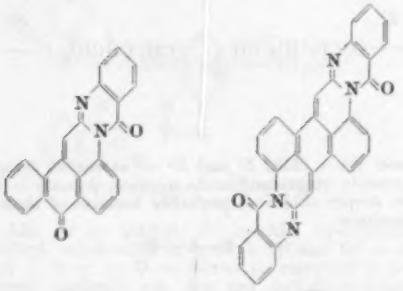
These are $A^1{\cdot}X{\cdot}A^2$, where A^1 and A^2 are the same or different optionally substituted anthraquinonyl or anthraquinone radicals (I) containing a heterocyclic ring fused so that both keto groups of I are still present, at least one, preferably both As, containing a hexahydrobenzoylamino group (II), and X = direct link or an aromatic or heterocyclic radical. They are vat dyes of good fastness, and dye natural and regenerated cellulose the same hue and have better levelling properties than the analogous dyes with benzoylamino instead of II. Thus, a mixture of 1,4-diamino - 2,2′ - (5′,6 - phthaloyl)benzothiazolylanthraquinone (5), hexahydrobenzoyl chloride (5), pyridine (3), and nitrobenzene (120) is heated at 150–155°C to give



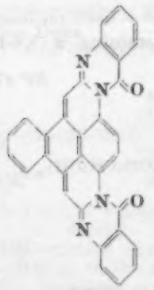
a very level-dyeing fast reddish blue vat dye. A.T.P.

Disperse Dyes for Polyesters and Polyamides
FBy *BP* 872,349 (Germany 10 Jan 1959)

Polyester and polyamide fibres are dyed in brilliant colours of excellent sublimation, light, and washing fastness by I, II, and III



(I) (II)

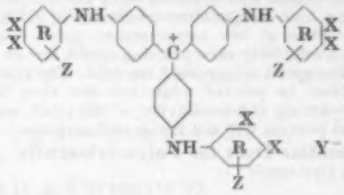


(III)

and by their substitution products. They are obtained by known methods. e.g. condensation of aminoanthraquinones with acetanthranils or of acetylaminoanthraquinones with anthranilic acids, in presence of dehydrating agents or inert solvents and molten $ZnCl_2$. Thus, 1-aminoanthraquinone (22·3) is heated at 100°C with acetic anhydride (200) (with removal of acetic acid) and, after addition of $ZnCl_2$ (25) and anthranilic acid (15), at 100–110°C for 2 h to give, on filtering, I, a brilliant greenish yellow dye of excellent fastness. A.T.P.

Triphenylaminotriphenylmethane Dyes
FH *BP* 872,561 (Germany 13 Oct 1956)

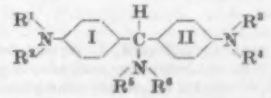
The above are



(one X = H, the other = OH, Alk, CF_3, Ar, Alkoxy, SO_3H, SO_2NH_2, COOH or ester, CN, CH–CH·COOH, $SO_2CH_2CH_2OH$ or halogen, Z = H, Alk or SO_3H, X and Z in each R are identical and in the same position, Y = Cl, Br, I, or OH or a complex of $AlCl_3$, $FeCl_3$, or $ZnCl_2$). They are obtained by reacting triphenylcarbinols (or their simple salts) containing *para* to the central C a halogen, OH, or alkoxy (at least one *para* group being halogen) and which may have further halogen or CH_3 in any free position and SO_3H *meta* to the central C, with the requisite aromatic amine. They are basic dyes and, when SO_3H is present, dyes for paper and inks. Because of the cheapness of starting materials and high yield and purity of the products, the process is more suitable for industrial use than are known methods. Thus, the $ZnCl_2$ double salt of 4,4′-dimethoxy-4″-chlorotriphenylchloromethane (25) is heated for 5 h at 145°C with *m*-chloroaniline (80). Excess of amine is removed and, on stirring into hot dilute HCl, the product, a blue powder, is obtained in 92% yield. A.T.P.

Bis-(*p*-Dialkylaminoaryl)methylamines
Allied Chemical Corp. *BP* 873,186 (U.S.A. 13 May 1958)

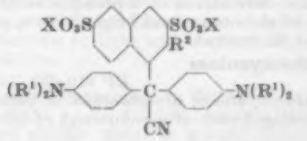
New derivatives of bis-(*p*-dialkylaminoaryl)methanes have improved solubility characteristics and stability, rendering them more useful for "colourless carbon papers", impact-printing papers, duplicating manifold printing systems, etc., than known compounds of this type. They are



(R^1 to R^4 = Alk of 1–5 C, R^5 = aliphatic radical, R^6 = H or aliphatic radical, rings I and II may contain halogen, alkyl, alkoxy, dialkylamino, acylamino or sulphonamido). They are obtained by condensing bis-(*p*-dialkylaminoaryl)-methanes containing a replaceable atom or group on the methane C atom with the appropriate aliphatic amine, or from *C,C*-bis(dialkylaminoaryl)methylamines and aliphatic compounds containing reactive groups, or by alkylation of *N*-bis(*p*-aminoaryl)methyl derivatives of aliphatic amines. Thus, 90% Michler's Hydrol (120), diethylamine (36·5), and toluene (500) are autoclaved at 100–105°C for 24 h, half the toluene is distilled off, and the product, *N*-bis(*p*-dimethylaminophenyl)methyl diethylamine, filtered off. Its toluene solution, when poured on to a surface coated with attapulgite clay, instantly develops an intense blue colour. A.T.P.

Dye Cyanides having a High Temperature-coefficient of Photochemical Reaction
L. Chalkley *USP* 2,951,855 (28 Aug 1958)

Hydrophilic dye cyanides of formula

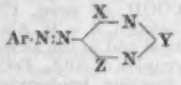


(R^1 = CH_3 or C_2H_5; R^2 = H or OH; X = cation) which in presence of an activator form a coloured compound on exposure to ultraviolet and shorter-wavelength radiation are obtained by treating the corresponding dye with an ionisable cyanide in aqueous solution and isolating the product either as the free acid or as a

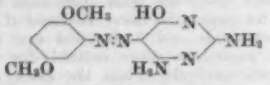
salt. Thus, an aqueous solution of Naphtnalene Green V (C.I. Acid Green 16) and NaCN, mixed at 80°C and allowed to cool by standing for 12 h, yields the colourless cyanide of the dye. These dye cyanides have the unique property of having a high temperature-coefficient of photochemical reaction. Thus at low temperatures paper coated with them has a relatively slow printing speed, but at 70–160°C the printing speed is increased ten-fold. The coated paper can therefore be printed while hot and then be cooled, thereby reducing the sensitivity of the print, so that the unexposed portion will not fog in ordinary use. C.O.C.

Azopyrimidine Dyes for Polyacrylonitrile
American Cyanamid Co. *BP* 871,624 (U.S.A. 11 Jan 1957)

Azopyrimidine dyes of formula



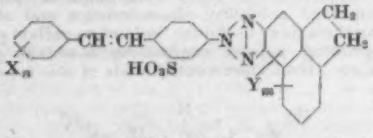
(Y = amino or alkylated amino; either X or Z or both = amino or alkylated amino) have excellent affinity for acrylonitrile polymers containing ≪ 85% of acrylonitrile and free from basic comonomers. Thus



applied at the boil to acrylic fibres from an aqueous bath containing acetic acid and Na acetate yields a reddish brown dyeing of good fastness to light, alkaline perspiration, washing, and rubbing. C.O.C.

Stilbene-Triazole Fluorescent Brightening Agents
HW *BP* 874,611 (9 Oct 1956)

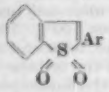
Compounds of formula



(X and Y = same or different, H or monovalent neither chromophoric nor auxochromic radicals; n = 1–5; m = 1–4) e.g. 2-(4″-styryl-3″-sulphophenyl)(acenaphtheno-6,7′-4,5)-1,2,3-triazole, are fluorescent brightening agents, particularly suitable for application to cellulose and polyamides. They are fast to chlorine and washing, and have good build-up properties. C.O.C.

Thiophen Dioxide Fluorescent Brightening Agents and Coloured Fluorescent Compounds
O. Dann *BP* 871,351 (Germany 17 Sept 1956)

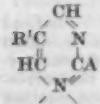
Compounds of formula



are fluorescent brightening agents for all types of fibres. Thus Terylene, boiled for 1 h in an aqueous dispersion of 2-phenylbenzothiophen-1,1-dioxide and rendered acid with formic acid, is given a high whitening effect which is very fast to light. The compounds are also applicable to many other substrates, e.g. plastics, leather, soap, etc. Some of the compounds are coloured: thus, the diamino and diacetylamino derivatives of 2-phenylbenzothiophen-1,1-dioxide are bright orange and bright yellow, respectively, and both are fluorescent. C.O.C.

Pentamethinecyanines
Ilford *BP* 870,633 (14 May 1958)

Pentamethinecyanines are obtained in yields of about 90% by treating 1 mol. of a compound of formula

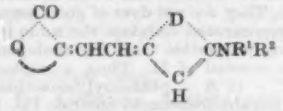


with one of formula

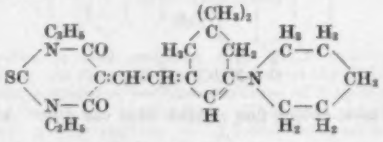
(R^1 = H, Alk, Ar or aralkyl; R^2 = Alk, Ar or aralkyl; R^3 = Alk, Ar, aralkyl, hydroxyalkyl or carboxyalkyl; A = H or a monovalent substituent; M = N or –CH=; X and Y = acid radicals, D = atoms to complete a 5- or 6-membered ring; n = 0 or 1). Thus 2 mol. of 2-methylbenzothiazole ethiodide and 1 mol. of 2-methylthiopyrimidine methiodide were dissolved in alcohol and refluxed in presence of triethylamine as catalyst to give a 91·2% yield of the green bis-2-(3-ethylbenzothiazole)-pentamethinecyanine iodide. C.O.C.

Hemioxonol Dyes containing a Carbocyclic Nucleus
Eastman Kodak Co. *USP* 2,956,881 (12 Mar 1957)

Dyes of formula



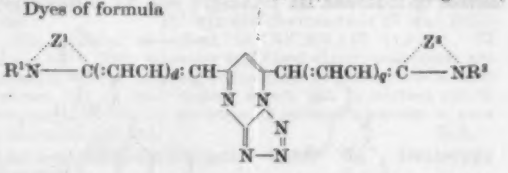
(R^1 and R^2 = Alk or Ar or together = atoms to complete a 5- or 6-membered heterocyclic nucleus; D = atoms to complete a cyclohexene ring; Q = atoms to complete an indandione or a 5- or 6-membered heterocyclic nucleus), e.g. the blue



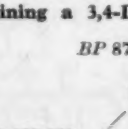
have photosensitising properties. C.O.C.

Undissociated Cyanine Dyes containing an Azaindenyl Nucleus
Kodak *BP* 871,113 (U.S.A. 30 Aug 1956)

Dyes of formula



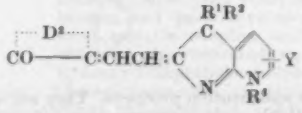
(R^1 and R^2 = Alk; Z^1 and Z^2 = atoms to complete a benzoxazole or benzothiazole nucleus; d and q = 0, 1 or 2) are deeply coloured, probably because of their mode of resonance



(A = acid nucleus; B = basic nucleus). C.O.C.

Cyanine Dyes containing a 3,4-Diazaindene Ring System
Ilford *BP* 870,753 (4 July 1957)

Dyes of formula



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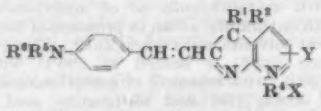
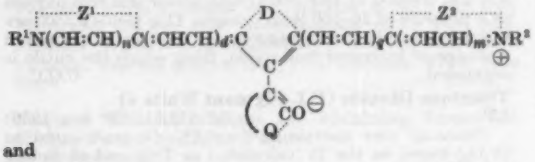


(R^1, R^2, R^5 and R^6 = Alk; R^3 = Alk, hydroxyalkyl or aralkyl; R^4 = Alk or aralkyl; n and m = 0 or 1; X = acid

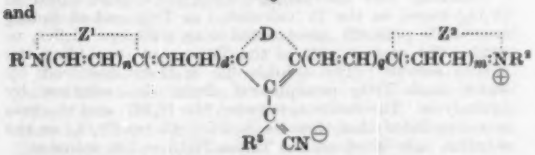
radical; Y = H or Alk; Z = CH or N; D^1 = atoms to complete a 5- or 6-membered ring; D^2 = atoms to complete a keto-methylene nucleus), e.g. the yellow (1,1,4-trimethyl-3,4 - diazaindene - 2)(3 - methyl - 2 - benzothiazole)methin - cyanine iodide, have photosensitising properties. C.O.C.

Holopolar Cyanine Dyes
Eastman Kodak Co. *USP* 2,955,939 (13 Sept 1956)

Dyes of formula



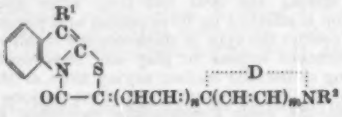
and



(R^1 and R^2 = Alk; R^3 = cyano or alkoxy; d, m, n, and q = 0 or 1; D = atoms to complete a cyclopentene or cyclohexene ring; Q = atoms to complete an indandione or a 5- or 6-membered heterocyclic nucleus; Z^1 and Z^2 = atoms to complete a 5- or 6-membered nucleus, e.g. 1,3-diethyl-5 - [2,5 - di(1 - ethyl) - 2(1*H*) - naphtho[1,2]thiazolylidene)-cyclopentylidene]barbituric acid, have excellent photosensitising properties and are best used dissolved in pyridine. C.O.C.

Polymethine Dyes
Ilford *BP* 874,809 (6 Feb 1959)

Dyes of formula



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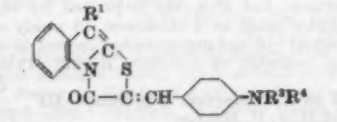
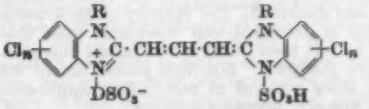


(R^1 = Alk, Ar or aralkyl; R^2 = Alk, hydroxyalkyl, carboxyalkyl, sulphoalkyl or aralkyl; R^3 and R^4 = Alk; n and m = 0 or 1; D = atoms to complete a 5- or 6-membered nucleus), e.g. the red 5-(3-ethyl-2-benzothiazolimylidene) - ethylidene - 3′ - methylindolo - (2′,1′:2,3) - thiazolid-4-one, have photosensitising properties. C.O.C.

Anhydrocarbocyanine Dyes
Kodak *BP* 871,936 (U.S.A. 23 Aug 1957)

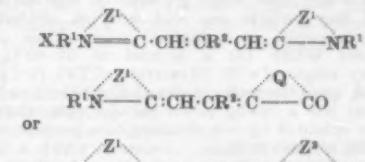
Dyes of formula



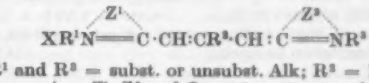
(R = Alk of 1–4 C; n = 1 or 2; D = alkylene), e.g. anhydro-5,5′,6,6′- tetrachloro - 1,1′- diethyl - 3,3′- di(4 - sulphobutyl)-benzimidazolocarbocyanine hydroxide, are particularly useful for sensitising, in the green region of the spectrum, silver halide emulsions containing magenta-forming colour couplers, e.g. 1-(2,4,6-trichlorophenyl)--3 - [3 - 2,4 - di - t - amylphenoxyacetamido) - benzamido] - 5 - pyrazolone, since they have little tendency to be displaced from the silver halide grains by the colour couplers. C.O.C.

Symmetrical and Unsymmetrical Carbocyanine and Merocyanine Dyes
Kodak and Kodak-Pathé *BP* 871,930 (France 3 Apr 1957)

Condensation of an orthoester with a cyclammonium quaternary salt containing a reactive group, when carried out in presence of a tricresyl phosphate or a phenol as reaction medium and an alkali-metal hydroxide or an organic tertiary amine having a dissociation constant greater than that of pyridine as basic condensing agent, results in good yields of dyes of formula



or



(R^1 and R^2 = subst. or unsubst. Alk; R^3 = H or Alk; X = anion; Z^1, Z^2 and Q = atoms to complete a 5- or 6-membered nucleus; R^1 is different from R^2 and/or Z^1 from Z^2), depending upon the starting materials chosen. C.O.C.

Polymethine Dyes containing a 4-(Hydroxymethyl)- or 4-(Acetoxymethyl)-Δ^2-thiazoline or Θ-oxazoline Nucleus
Gevaert Photo-Producten *USP* 2,954,376 (8 Apr 1958)

Gives general formulae for, and 24 examples of, cyanine, styryl, merocyanine, and rhodacyanine dyes of the type listed in the title. C.O.C.

Presscakes having Increased Pigment Content
Gy *BP* 871,180 (U.S.A. 23 July 1958)

A solution of a methylcellulose or methyl starch which is soluble in cold water but not in hot, is added to an aqueous suspension of the pigment (> 2% methylcellulose or methyl starch on the dry wt. of pigment). The dispersion is then heated until flocculation occurs and is filtered at or above the temperature of flocculation. This results in presscakes containing 35–55%, on their dry wt., more pigment than those prepared without use of the above assistants. C.O.C.

Phthalocyanine Pigments
Gy *BP* 872,659 (14 July 1958)

Microcrystalline phthalocyanine pigments of good colour strength and which do not crystallise in aromatic solvents are obtained by milling phthalocyanines (I) with a salt of an organic or inorganic acid which is inert to and harder than (I) and which is easily soluble in an organic or inorganic solvent in presence of 10–50% of pyridines substituted by alkyl or cyclically bound alkenylene and optionally by other non-ionogenic groups. The pigments are of β-crystal phase and have excellent tinctorial strength, and dye lacquers, resins, synthetic-polymer fibres, rubber, and paper. Thus, Cu phthalocyanine (II) (C.I. 74160) (30), dehydrated $CaCl_2$ (90), quinoline (4·5), and glycerol mono-oleic acid ester (1·5) are ball-milled for 4 h, the temperature being allowed to rise to 50°C. The milled material is stirred for 1 h at 80–85°C in 2% HCl (700) and filtered to give a blue pigment of greener shade than II dissolved and reprecipitated by known methods, and showing no crystal formation on boiling for 2 h in toluene. A.T.P.

Mixtures of Cobalt Phthalocyanine and Cobalt Phthalocyanine Phenylsulphones— Bright Blue to Greenish Blue Vat Dyes
General Aniline *USP* 2,953,574 (20 Dec 1953)

A mixture of cobalt phthalocyanine and a cobalt phthalocyanine in which 1–4 of the benzene rings are substituted by a substituted or unsubstituted phenylsulphonyl group is a bright blue to greenish blue vat dye of excellent fastness to light and washing, and improved fastness to chlorine. C.O.C.

Metal Pigments Dispersible in Water
Walter Marx & Co. *BP* 870,783 (Austria 19 Nov 1958)

A powdered ductile metal is ground while water is added. Thus atomised aluminium (C.I. Pigment Metal 1) of particle size < 500 mμ (30–150 parts by wt.) is ball-milled with stearic acid (20–75) as lubricant, and casein (0·1–1·5) as emulsifying agent, and water. After grinding is complete, the product is washed with water and brought to the consistency of a paste by pressing. C.O.C.

Mineral Filler especially useful with Titanium Dioxide (C.I. Pigment White 6)
Soc. Anon. du Blanc Omya
BP 872,149 (France 2 Sept 1957)

A mineral filler of particle size 5–7 μ is treated in absence of water with white pigment of high refractive index and fine particle size and with an aliphatic or aromatic carboxylic or sulphonic acid. Thus calcite (C.I. Pigment White 18) is ground at 50–80°C with 1·5–2·0% by weight of a 50 : 50 mixture of TiO_2 (0·1 μ) and stearic acid, until the particle size of the calcite is 5–7 μ. The product has a hiding power greater than that of a similar one obtained by wet grinding to a particle size of 1–3 μ and it is more brilliant. Paints in which it is used yield films of high hiding power, great hardness, and excellent resistance to ageing. C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7)
Columbian Carbon Co. *USP* 2,957,755 (11 June 1957)

A method is described in which a hydrocarbon is dispersed in a turbulent stream of hot blast-furnace gases at a temperature above that at which the hydrocarbon is decomposed into Carbon Black. It is especially suitable for production of the FF grade of pigments for use with rubber. C.O.C.

High-structure Carbon Blacks (C.I. Pigment Black 6 and 7)
Columbian Carbon Co. *BP* 870,720 (U.S.A. 21 Aug 1958)

A method is described in which highly aromatic oils, e.g. creosote and coal tar oils, are used as the raw material. C.O.C.

Improving the Quality of Carbon Black Pellets (C.I. Pigment Black 6 and 7)
Phillips Petroleum Co. *USP* 2,953,437 (21 Dec 1956)

Treating either wet or dry pellets containing fines with a Carbon-Black-furnace off-gas for a short period under conditions which suspend the pellets in the gas, reduces the fines content, lowers the modulus, and reduces the moisture content. C.O.C.

Nacreous or Pearlescent Basic Lead Carbonate
Francis Earle Laboratories *USP* 2,950,981 (10 Apr 1957)

The product obtained by precipitating crystals of basic lead carbonate (C.I. 77597) from solutions of PbO in organic acids of higher mol.wt. than acetic acid, e.g. propionic, β-chloropropionic or iso- or n-butyric acid, has a lustre at least equal to that of natural Pearl Essence (C.I. Natural White 1). C.O.C.

Red Mercury–Cadmium Sulphide Pigments
Glidden Co. *USP* 2,950,984 (U.S.A. 26 July 1957)

The pigment obtained by mixing a solution of mercury in nitric acid with a solution of an alkali- or alkaline-earth-metal sulphide and then with a solution of a cadmium salt is much improved in physical and chemical properties if the slurry of the coprecipitated mercury and cadmium sulphides is treated with enough sodium silicate and/or aluminate to inhibit peptisation and/or oxidation of the pigment in subsequent processing. C.O.C.

Titanium Dioxide (C.I. Pigment White 6) from Titanium Tetrachloride
American Cyanamid Co. *USP* 2,957,753 (5 Nov 1958)

Describes manufacture of both anatase and rutile by combustion of a homogeneous stream containing $TiCl_4$ and O_2. The process, which is continuous, and the apparatus are very simple, and no separate nucleating agent is necessary. C.O.C.

Rutile (C.I. Pigment White 6) of Improved Fastness to Light
National Lead Co. *BP* 874,511 (Germany 22 Mar 1958)

An aqueous slurry of rutile is treated with a water-soluble silicate, an aluminium salt and a cerium salt, and then with alkali until the mixture is neutral or alkaline, so as to produce a coating of a Si, an Al, and a Ce compound on the pigment. The treated pigment has improved fastness to light and greater resistance to chalking. It is suitable for use with plastics, paper laminates, textiles and leather, and for brightening coloured pigments. C.O.C.

Production of Rutile (C.I. 77891) from Ilmenite and related Ores
Commonwealth Scientific and Industrial Research Organization *BP* 872,944 (Australia 31 Oct 1956)
A. J. Gaskin and A. E. Ringwood
USP 2,954,278 (Australia 31 Oct 1956)

The ore is heated at 600–1000°C in absence of oxygen and presence of free sulphur vapour, cooled in absence of air, and treated at 100–200°C with water and free oxygen at a pressure of 20–100 lb/in² gauge. This yields a mixture of granular skeletal rutile aggregates, globular sulphur, and a sludge of hydrated iron oxide, from which the rutile is separated. C.O.C.

Titanium Dioxide (C.I. Pigment White 6)
LT *BP* 873,611 (30 Aug 1956)

Titanium ores containing > 0·5% Cr (calculated as Cr_2O_3) based on the Ti (calculated as TiO_2 and excluding any rutile present), are treated with sufficient H_2SO_4 to render the greater part of the Ti present (excluding the rutile) soluble. The soluble Ti is then dissolved by water and TiO_2 precipitated from the solution by hydrolysis. The reaction between the H_2SO_4 and the ores is so regulated that there is ≯ 0·5% Cr (as Cr_2O_3) in the solution calculated on the Ti (as TiO_2) in the solution. C.O.C.

Stabilised Aqueous Solutions of Direct Dyes (VIII p. 523)
Photochemical Printing and Compounds therefor (IX p. 525)

V—PAINTS; ENAMELS; INKS

Surface Drying of Linseed Oil Emulsion Paints
A. W. Schwab, W. L. Kubie, J. A. Stolp, J. C. Cowan, and H. M. Teeter *Off. Dig. Fed. Soc. Paint Technol.*, 33 (June 1961) 747–758

Surface drying of linseed oil emulsion paints has been observed during the first half-hour after application. Drying time is affected by formulation and pigment-to-oil ratio, but neither the type of thickener nor small variations in water content appear to play any important part in determining either the surface-drying time or the water-sensitivity of the non-ionic paint formulations studied. Films from the ionic formulations examined had poor water-resistance, but this was improved by the use of hydroxyethylcellulose as a thickener. A study of drying curves of pigmented and unpigmented linseed oil emulsions revealed no evidence of inversion during drying. J.W.D.

Zinc Dust as a Protective Pigment—III
A. Pass and M. J. F. Meason
J. Oil & Col. Chem. Assocn., **44** (July 1961) 417–441

Possible mechanisms by which zinc-rich paints protect steel under immersion conditions are discussed. These include barrier or resistance protection, electrochemical cathodic protection, and the deposition of basic Zn cpd. resulting partly from interactions of the corrosion products from the electrochemical reactions. This deposited film finally hardens into a strong and apparently impervious layer giving protection over several years. The paint and application properties required for these mechanisms to operate are described. The toxicity of Zn towards marine organisms is discussed and compared with that of Cu. Results of raft tests on zinc-rich paints based on organic media are discussed. Zinc-rich paints bound with alkali silicates show promise in anti-fouling applications when used on properly prepared surfaces. J.W.D.

Gas Evolution in Zn (C.I. Pigment Metal 6) Paints
W. L. Lantz
Paint Varnish Production, **51** (3) (1961) 25–30, 80
Chem. Abs., **55** (26 June 1961) 12881

Gassing is caused by presence of water and/or fatty acids, and can be prevented by incorporating a "water scavenger" e.g. $(C_2H_5)_4SiO_4$, and neutralising agents, e.g. diethylenetriamine or CaO. C.O.C.

Effects of Radiation, Humidity, and Atmospheric Conditions on the Mechanical Properties of Paint and Varnish Films
H. W. Talen
Paint Varnish Production, **51** (2) (1961) 25–42
Chem. Abs., **55** (26 June 1961) 12881

Tensile tests of detached films exposed to the above agencies and to outdoor weathering were periodically

made on a variety of clear and pigmented compositions. These data also yielded stress–strain curves, elongation at break, and modulus values. This method gave significant results in much less time than conventional outdoor exposures. The ageing of alkyd coatings is a stiffening process caused by macromolecular growth. Ageing of oleo-resinous and bodied-oil varnishes is caused by a balance of progressive oxidation and polymerisation. ZnO (C.I. Pigment White 4) reacts with vehicles in both the fluid paint and the dry film. Non-chalking rutile TiO_2 (C.I. Pigment White 6) is moderately reactive with alkyd vehicles, whereas anatase TiO_2 (C.I. Pigment White 6) is inert. C.O.C.

Sodium Silicoaluminates for obtaining Improved Hiding Power in Latex Paints
R. R. Browning
Paint Varnish Production, **50** (13) (1960) 39–42
Chem. Abs., **55** (26 June 1961) 12881

Na silicoaluminate pigments prepared by treating Al salts with alkali silicates are efficient extenders when used with rutile TiO_2 (C.I. Pigment White 6) in aqueous systems. They result in better dispersion and hiding power. Their presence reduces the tendency of the rutile to reagglomerate when agitation stops. C.O.C.

Effect of γ- and Neutron-radiation on the Properties of a Coat of Varnish
Z. Jedlinski, Z. Hippe, and T. Uminski
Przem. Chem. No. 2 (1960) 110–112
Polish Tech. Abs., No. 3/4 (39/40) (1960) 159–160

Radiation from radium and beryllium affects the ageing process. Irradiation by radium (100 mC) in a beryllium container improves the impact resistance of coatings containing polyvinyl butyral, copolymer of vinyl chloride and the isobutyl ether of polyvinyl alcohol, modified alkyd resins, or polymerised linseed oil. Loss of elasticity and of adhesive power occurs, however, in coatings containing chlorinated rubber (polychlor), chlorinated polyvinyl chloride, phenol–formaldehyde resins, and modified tung oil. R.A.

Coatings Based on Acrylamide Interpolymers
H. A. Vogel and H. G. Bittle
Off. Dig. Fed. Soc. Paint Technol., **33** (June 1961) 699–708

The performance of acrylamide interpolymers in several formulated enamels based upon several different interpolymer resins is discussed and comparisons are made with alkyd–amine enamels. Finishes based upon these polymers can be varied in properties to cater for a wide range of end-uses by (*a*) choice of the monomer used with the acrylamide, and (*b*) use of various modifying resins added to the interpolymers. J.W.D.

Thermosetting Compositions Based on Acidic Copolymers Cross-linked with Diepoxides
J. D. Murdock and G. H. Segall
Off. Dig. Fed. Soc. Paint Technol., **33** (June 1961) 709–718

The development of enamels based on acidic copolymers (e.g. styrene–methyl acrylate–acrylic acid) cross-linked by means of diepoxides is described. The properties emphasised are adhesion to metal, appearance, application properties, and resistance to hot detergents and to grease. Copolymers of high styrene or vinyltoluene content have high resistance to detergents at elevated temp. The most useful enamels were found to be those based on copolymers having mol.wt. of ca. 30,000 containing 8% acrylic acid, and cross-linked with bisphenol–epichlorohydrin condensates. J.W.D.

PATENTS

Erasable Graphitic Ink
Venus Pen & Pencil Corpn. *USP* 2,956,038 (12 Dec 1958)

An ink suitable for ball-point pens consists of fine crystalline flake graphite (C.I. Pigment Black 10) of particle size 0·5–5·0 μ, preferably 2–4 μ, dispersed in white mineral oil of medium viscosity, e.g. about 69 Saybolt seconds at 210°F, in presence of a resin soluble in the oil and which does not separate from it when the ink is applied to paper, e.g. the polymono- and polydi-olefins from cracked petroleum. C.O.C.

Driers
Esso Research & Engineering Co.
USP 2,955,949 (17 Sept 1958)

The Co, Mn, and Pb salts of C_9 to C_{20} branched-chain carboxylic acids of which $\leqslant$ 75% is an acid of formula $R^1R^2R^3COOH$ (R^1, R^2, and R^3 = same or different Alk) have outstanding drying properties, excellent solubility and compatibility with paint vehicles, and little or no odour. C.O.C.

Aluminium Hydrocarbon Compounds for Curing Oxidised Drying Oils
Esso Research & Engineering Co.
USP 2,955,950 (26 Nov 1958)

Thick films (> 3 mils) of oxidised drying oils set to a hard coating in a very short time at room temperature if the drier used is a compound of formula R^1R^2AlX (R^1 and R^2 = Alk; X = Alk, H or Hal), e.g. Al triethyl, Al diethyl chloride or di-isobutyl Al hydride. C.O.C.

Pigmented Synthetic Resin Lacquers Dispersible in Aqueous Media
Ciba *BP* 870,460 (Switzerland 18 Feb 1957)

Lacquers readily dispersible in water, and which can be stored without sediment forming, are obtained by dispersing an inorganic pigment in a lacquer containing a hardenable water-insoluble etherified methylol compound of amide character capable of forming a hardenable aminoplast with HCHO and an organic solvent of at most limited miscibility with water. An ethylene oxide condensate of a water-insoluble organic compound containing at least one reactive H atom is used as the dispersing agent. In most cases the products are thixotropic and are viscous after being stirred up. C.O.C.

Epoxy-modified, Water-soluble Alkyd Resins becoming Hydrophobic when Cured
Rinshed-Mason Co. *BP* 870,412 (U.S.A. 28 Apr 1958)

Alkyd resins first modified by an epoxy resin and a monobasic acid and then treated with a base yield water-soluble products which become hydrophobic upon curing and have improved properties of adhesion and toughness. They have wide uses in water-thinned paints and inks. C.O.C.

Coating Compositions containing Pigment Metals
Aluminium Laboratories *BP* 873,232 (18 Apr 1958)

Aluminium (C.I. Pigment Metal 1) is readily dispersed in aqueous dispersions of film-forming agents by use of an oil-soluble, water-insoluble surfactant, a "coupling agent", e.g. an alcohol, and an auxiliary emulsifying agent, e.g. a fatty-acid soap of a volatile base. Films formed from the composition have good permanence and durability. C.O.C.

Colourless Fluorescent Coating Composition adapted to Overprinting
Sterling Drug *USP* 2,956,027 (10 Dec 1956)

A colourless varnish which can be overprinted on coloured surfaces to yield goods having in the printed areas the same colour in daylight and under ultraviolet radiation contains three compounds which fluoresce red, blue, and yellow respectively under ultraviolet radiation, all these compounds being colourless in 0·01–1·0% solutions. The preferred compounds are 2-(3-hydroxy-2-naphthyl)-benzimidazole (the only available red-fluorescing dye which gives a colourless solution in daylight), the blue-fluorescing 4-methyl-7-dimethylaminocoumarin, and the yellow-fluorescing 3,7-bis(phenylcarbamylamino)dibenzo-thiophen-5,5-dioxide. C.O.C.

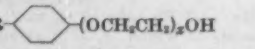
Organopolysiloxane Beaten Enamels Resistant to High Temperatures
Société des Usines Chimiques Rhône-Poulenc
BP 872,411 (France 5 Aug 1958)

Beaten enamels resistant to heat at 200–300°C comprise a methylphenylpolysiloxane of mol.wt. $\leqslant$ 4000 and a $(CH_3 + C_6H_5)$:Si ratio of 1·3–1·6 and C_6H_5:Si of 0·4–0·7 as binder, non-film-forming Al powder (C.I. Pigment Metal 1), a dimethylpolysiloxane gum of mol.wt. 100,000–500,000, and a solvent. C.O.C.

Universal Tinting Paste Vehicle
W. A. Cleary Corpn. *BP* 872,302 (Canada 11 Feb 1957)

A liquid tinting paste vehicle compatible with both organic and aqueous solvent paint systems contains

75–90% of linseed oil and 25–10% of a non-ionic surfactant of formula



(R = octyl or nonyl; x = 3–10). C.O.C.

Coating with Pigmented Polymers
Phillips Petroleum Co. *BP* 872,795 (U.S.A. 13 June 1958)
Powdered pigment is dispersed directly in a fluidised bed of polymer, e.g. polyethylene, and then the hot article to be coated is dipped into the bed while maintaining dense-phase fluidised conditions. Although the density and/or particle size of the pigment may not be the same as those of the polymer, uniform dispersion of the pigment is obtained with a resulting smooth and evenly coloured coating. C.O.C.

Light Stabiliser for Halogeno Resins (III p. 506)
Mineral Filler especially useful with Titanium Dioxide (C.I. Pigment White 6) (IV p. 516)
Letterpress Printing on Plastics and Inks therefor (IX p. 525)
Hexahydrophthalic Anhydride in Alkyd Resins (XIII p. 531)
New Types of Linoleum (XIII p. 532)
Coloured Mineral Granules (XV p. 536)

VI— FIBRES; YARNS; FABRICS

Cyanoethylation of Cotton
H. Friedrich *Melliand Textilber.*, **42** (June 1961) 657–662
Two- and single-bath and alkali-salt cyanoethylation procedures and, in particular, a new impregnating process for cotton are discussed. The influence of the individual factors concerned is considered, viz., fibre, caustic soda, moisture content of fibre, activity of acrylonitrile, temp., and time in two-bath process, and formation of dicyanodiethyl ether. Technical effects obtained by cyanoethylation are described, viz., mechanical strength, stability towards rot and heat, and behaviour towards acids, alkalis, and washing. Finally the problem of dyeing of cyanoethylated cotton is discussed. There are 16 references. H.H.H.

Effects of Various Spinning Processes on the Fine Structure of Viscose Rayon Staple
T. Kōshiro and T. Fujimura *Bull. Chem. Soc. Japan*, **34** (Apr 1961) 476–480 (*in English*)
Oxidising hydrolysis has been applied to the study of various types of rayon— spun in conc. sulphuric acid, dil. sulphuric acid, high zinc percentage Müller's bath, etc. Samples were refluxed at 100 ± 1°C for 30 min with a mixture of 4-N H_2SO_4 and 0·3-N potassium dichromate. Chain-length distribution and carboxylic acid content were determined. Tyre-cord, high-tenacity, Toramomen, and ordinary rayon exhibit only one peak in the crystallite-size distribution curves, whereas Lilienfeld rayon shows two such peaks. In the latter type the crystallites are larger. Treating Lilienfeld rayon with alkali results in an increase in crystallinity and in crystallite size. The carboxylic acid content of the oxidising-hydrolysed residue increases rapidly in the early stages of hydrolysis, probably owing to preferential attack in the amorphous region, but then decreases to an equilibrium value. I.G.L.

Tricel— Its Development, Manufacture, Properties, and Uses
M. W. Alford *J. Textile Inst.*, **52** (June 1961) P242–P255
A historical account of the development of Tricel is followed by details of its manufacture and preparation of modified and blended yarns. Of the properties of Tricel, setting is given special attention, and the paper concludes with a summary of its uses. P.B.S.

Sequence of Amino-acid Residues in Proteins
F. Sanger *J. Polymer Sci.*, **49** (Jan 1961) 3–29
A review of the methods available for the study of the sequence of amino-acid residues in proteins. P.T.S.

Peptide Synthesis and Protein Structure
P. G. Katsoyannis *J. Polymer Sci.*, **49** (Jan 1961) 51–74
The paper describes how peptide chemistry can assist the protein chemist in the structural analysis of proteins. A critical analysis is made of the reliability of the techniques of determination of amino-acid sequence in proteins. P.T.S.

Phase Changes in Proteins and Polypeptides
P. J. Flory *J. Polymer Sci.*, **49** (Jan 1961) 105–128
An analysis is given of permitted chain arrangements in crystalline polymers, including proteins, and the transition from crystalline to amorphous forms. The kinetics of the transition are examined, and it is shown to resemble a phase transition. P.T.S.

Use of Rotatory Dispersion in Determination of Protein Structure
J. A. Schellman and C. G. Schellman
J. Polymer Sci., **49** (Jan 1961) 129–151

Use of X-Ray Diffraction in Determination of Protein Structure
B. M. Low *J. Polymer Sci.*, **49** (Jan 1961) 153–175

Structure of Fibres of the Collagen Group and Related Matters
W. T. Astbury
J. Soc. Leather Trades Chem., **45** (May 1961) 186–214
A detailed discussion is given of present evidence based on X-ray analysis of fibres of the collagen group. It is postulated that the mammalian collagens approximate to an ideal limiting structure, for which the name "eucollagen" is proposed, in which glycine occurs at intervals of three residues and hydroxyproline every ten residues. Glycine and hydroxyproline thus provide the characteristic internal linkages of the configurational unit revealed by X-rays; the apparent conflict between the findings of the physical X-ray test and the chemical hydroxyproline test is thus resolved. The structures of tropocollagen and elastin are also discussed in detail in the light of the foregoing theory. Elastin is shown to be a member of the collagen group that has been adapted to bring its thermal shrinkage temp. below ordinary temp. The bulky valine residue, inserted in the only place where hydroxyproline could be accommodated, with its stabilising effect on structure, prevents any approach to a crystalline structure and induces elasticity. Biological calcification and the biogenesis of collagen are also treated in detail. J.W.D.

Studies on Cystine in Wool

VII— Conditions for the Estimation of SH Groups in Wool by the P-CMB Method
Y. Nakamura and Y. Nemoto
J. Soc. Textile Cellulose Ind. Japan, **17** (May 1961) 428–435
A method for differentiating between and determining free SH groups and those combined in cysteine residues in wool treated with thioglycollic acid has been developed and full details are given. The Human's polarographic method has been employed using *p*-chloromercuribenzoic acid (P-CMB).

VIII— Polarographic Estimation of SH Groups in Wool Treated with Thioglycollic Acid
Y. Nakamura, Y. Kadoya, and Y. Nemoto
Ibid., 435–440
Estimations of the SH content of wool treated with thioglycollic acid have been carried out as outlined in VII, and differences in the action of the thioglycollic acid in acidic and alkaline media investigated. (1) The amount of mercaptide and P-CMB adsorbed (except P-CMB taken up by reaction with the combined SH in wool) decreases with increase in the pH of the P-CMB solution, as does the ratio of adsorbed mercaptide to adsorbed P-CMB. (2) The extent of disulphide-bond rupture on reaction with P-CMB increases with rise in pH of the wool–thioglycollic acid treatment, particularly under acid conditions. (3) Reaction of wool with P-CMB cannot be carried out at low pH. (4) The amount of thioglycollic acid taken up by wool is dependent on pH, being greater in alkaline medium than with thioglycollic acid alone. In the former case the detected SH content is nearly equal to the combined SH. (5) Presence of another anion reduces the amount of thioglycollic acid adsorbed by wool except at pH > 7. Decrease in the SH content of wool treated with thioglycollic acid by rinsing is more marked in acidic than in basic media. I.G.L.

Felting and Tip Weathering of Wool

G. F. Flanagan *Text. Research J.*, 31 (June 1961) 576–577

Describes the results of work which support the unequal-migration-speed theory of felting and offer an explanation of the results described by Martin (J.S.D.C., 60 (1944) 325–328). C.O.C.

New Fibre Developments

C. E. Coke *Canadian Textile J.*, 78 (9 June 1961) 39–44

A brief review of production of modified natural and synthetic fibres over the past sixty years. Emphasis is placed on the physical properties of existing fibres and the commercial possibilities of fibres such as nylons 7 and 12. F.J.

Polyamide 11—Rilsan

P. A. Koch *Tinctoria*, 58 (May 1961) 189–194

Table of properties including definition and trade names; discovery and development; raw materials; manufacture; chemical constitution; physical and chemical properties; types of product; applications; patents; bibliography. E.V.T.

Proton Magnetic Resonance of Some Polyamides

A. E. Woodward, R. E. Glick, J. A. Sauer, and R. P. Gupta *J. Polymer Sci.*, 45 (Aug 1960) 367–377

Plots of line width and second moment vs. temperature have been obtained for polyhexamethylene adipamide and polyhexamethylene sebacamide, dry and saturated with water and deuterium oxide. The transition temperatures determined by this method agree fairly closely with results from dynamic mechanical methods. P.T.S.

Polycaprolactam Fibre Crystallinity Determined by X-Ray Differential Filtration Method

G. W. Urbańczyk *J. Polymer Sci.*, 45 (July 1960) 161–168

Changes in Density of Steelon Filament in Relation to Draw Ratio and Heat Treatment

A. Wlochowicz *Przeg. Wlok.*, No. 8 (1959) 410–414
Polish Tech. Abs., No. 3/4 (39/40) (1960) 217, 219

The density of Polish Steelon rope, measured pycnometrically, was found to increase with the draw ratio. The density is lowest for raw, undrawn fibre and changes from 1·1319 g/cm³ at 0% to 1·1502 g/cm³ at a draw ratio of 4·75. Pycnometric methods were found to be reliable. The method can be applied to the control of production processes since both the degree of crystallisation and the degree of orientation can be obtained from density measurements. R.A.

Studies on Nylon 6 Fibres Irradiated by γ-Rays. I—Properties and Viscosity-average Molecular Weight of Undrawn Fibres Irradiated in Various Atmospheres

M. Hashimoto and H. Amemiya *J. Soc. Textile Cellulose Ind. Japan*, 17 (May 1961) 446–450

The effects of irradiating undrawn nylon 6 fibres in various atmospheres (air at 760 mm Hg, air at 10^{-3} mm Hg and nitrogen at 760 mm Hg) on viscosity, melting point, draw ratio at break, breaking strength, and solubility have been investigated. Irradiation at doses of up to 10 Mr results in a rapid decrease in the viscosity, but above 10 Mr the decrease is less marked. No linear relationship between viscosity and irradiation dose has been established. The breaking strength and draw ratio at break decrease in a similar manner. The solubility in 80% aq. formic acid is hardly affected by irradiation up to 10 Mr but increases rapidly on irradiation at doses from 10 to 110 Mr. At lower doses slight increases are caused in melting point, mechanical properties, and resistance to solvents, owing to cross-linking. These changes are dependent on the atmosphere in which irradiation takes place and indicate that the cross-linking and main-chain breakdown accompanying irradiation are due to the presence of oxygen, even in extremely small amounts. I.G.L.

Cause, Effect, and Prevention of Pilling

J. Nüsslein *Textil Praxis*, 16 (May 1961) 447–451

Pilling may be combated by using long-staple fibres, high yarn twist, thermosetting, or increasing the coefficient of friction of the fibres. Polyester fibre–wool blends may be rendered suitable for carded-yarn fabrics by partial decomposition with ammonia. J.W.D.

Polypropylene Fibres

A. B. Thompson *Man-made Text.*, 38 (July 1961) 35–38

A review of the properties, including the dyeing properties, of these fibres. C.O.C.

Production, Properties and Processing of Polypropylene Fibres

E. Bobek *Textil Praxis*, 16 (May 1961) 444–447

A survey, including a list of producers of polypropylene. J.W.D.

PATENTS

Regenerated Cellulose Filaments of Improved Whiteness

American Enka Corpn. *USP* 2,950,207 (7 Nov 1957)

A permanent white is obtained by incorporating in the spinning solution a mixture of a white inorganic pigment with a blue and a violet organic pigment. C.O.C.

Pigmented Rayon

VEB Sächsisches Kunstseidenwerk "Siegfried Radel" *BP* 872,207 (6 Jan 1958)

Pigments containing an anionic dispersing agent are added to the spinning solution, there being added either to the pigment or to the spinning solution, before the two are mixed, 0·01–0·25% (on wt. of spinning solution) of a condensate of formula $A(CH_2{\cdot}CH_2O)_xCH_2CH_2OH$ (A = RCH_2O, RC_6H_4O, RNH or RCOO (R = Alk of 5–50 C; $x > 1$). This gives much more uniform dispersion of the pigment and also better spinning properties to the solution. C.O.C.

Flame-resistant Cellulosic Compositions

Monsanto Chemical Co. *USP* 2,952,880 (30 June 1951)
USP 2,953,463–4 (30 June 1951)

Addition to an aqueous solution of cellulose of 3–25% (on wt. of cellulose) of the alkaline or neutral product obtained by reacting phosphoryl chloride and ammonia renders fibres, films, etc. produced from the solution flame- and afterglow-resistant. Such solutions can be used to produce fibres or films or as impregnating or coating agents for paper or textiles. C.O.C.

Increasing the Affinity for Dyes of Linear, High-molecular-weight Poly-α-olefins

Montecatini Societa Generale per l'Industria Mineraria e Chimica *BP* 872,432 (Italy 10 Jan 1958)

The affinity for dyes of polypropylene or ethylene–propylene copolymers, both of which contain peroxidic groups, is increased by grafting on to them polymeric chains derived from an isopropenylpyridine. Thus, polypropylene fibre containing 0·08% of hydroperoxide (–OOH) group is treated for 7 h at 80°C in a mixture of equal weights of 3-isopropenylpyridine and methyl-methacrylate. It is then washed with 4% H_2SO_4 at 90°C, then with boiling 1:1 benzene–acetone mixture, and finally with ether. The product dyes well with acid dyes. C.O.C.

Readily Dyeable Polyolefins

Montecatini Societa Generale per l'Industria Mineraria e Chimica *BP* 873,830 (Italy 12 Dec 1956)

A crystalline polyolefin, especially isotactic polypropylene, has mixed with it one or more synthetic polymers which are either soluble in or emulsifiable with molten polyolefin, have mol.wt. $\leqslant$ 1000, a second-order transition temperature $<$ m.p. of the polyolefin, are water-insoluble (or become insoluble during spinning) and have affinity for acid or disperse dyes. Thus polypropylene mixed with polyethylenediamine yields fibres having good mechanical properties and dyeing well with acid dyes. C.O.C.

Coloured Polyamides

Organico *BP* 872,558 (France 31 July 1956)

The above are formed by reacting polyamides or polyamide-forming reactants before or during their condensation with 0·1–5% of a halogenated amino or nitro derivative of an aromatic diacid having two CO groups in *ortho* positions, or with a halogenated, nitrated, aminated, or hydroxylated derivative of an aliphatic diacid having two CO groups in α, β, γ, δ or ε positions, or with a complete or partial anhydride or ester of such acids. The products are fluorescent yellow-green to orange-red and are stable to heat and the usual chemical agents. Thus, a mixture of 4-chlorophthalic anhydride (5) and nylon 6.6 granules (1000) is melted together to give an

intensely yellow fluorescent mass which may be moulded or spun without colour variation. A.T.P.

Light Stabiliser for Halogeno Resins (III p. 506)
Relative Wet-strength of Man-made Fibres and Yarns (VII this page)
Modifying the Dyeing Characteristics of Textile Fibres (VIII this page)
Migration of Liquids in Textile Assemblies. IV— Penetration of Fabrics by Liquids (X p. 526)
Adsorption of Iodine by Nylon 6 (XIII p. 531)
Stabilisers for Acrylonitrile Copolymers (XIII p. 532)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Relative Wet-strength of Man-made Fibres and Yarns

G. Klust *Textil Praxis*, **16** (May 1961) 514–516

Relative wet-strength (% of dry-state strength) of polyester, polyethylene, polyvinyl alcohol, polyvinyl chloride, and polyacrylonitrile monofilaments and yarns are given. The different effects of distilled water, chlorinated tap water, brackish water, and seawater are tabulated and briefly discussed, and the effect of immersion time is similarly treated. J.W.D.

Behaviour of Synthetic-polymer Fibres toward Carbonisation— II

G. von Hornuff and H. Hiller *Melliand Textilber.*, **42** (June 1961) 684–689

It had previously been shown (cf. *ibid.*, **41** (1960) 474) that polyamide fibres could not be carbonised with HCl gas without severe damage, and the use of aluminium chloride is now investigated. The results are discussed and the consequences resulting for the practical textile finisher are indicated. 17 References. H.H.H.

Energy of Interaction between Textile Fibres and Solid Soil in the Washing Process

H. Lange *Amer. Dyestuff Rep.*, **50** (12 June 1961) 433–436

Translation of article originally in *Kolloid Z.*, **154** (No. 2) (1957) 103–110; **156** (No. 2) (1958) 108–113. Theoretical calculations have been made of the energy of interaction between fibre and soil, and the application of the results to the washing process has been considered. Experimental work has indicated that the hydration layers surrounding the fibres and the soil particles are particularly important for the washing power of non-ionic detergents, because these agents cannot increase the electrical repulsion between fibre and soil particles. W.G.C.

Use of Modern Home Laundry Equipment. I— Wrinkling Effects on Swatches of Fabrics

R. K. Taube, E. S. Ross, and N. D. Poole *Amer. Dyestuff Rep.*, **50** (26 June 1961) 468–479

This study of laundering procedures investigates wrinkling effects on swatches of 27 different fabrics of cellulose acetate, cotton, acrylic, polyamide, polyester fibres, and some blends of these, through nine launderings. Subjective judgments of the need for ironing of fabrics were used throughout to evaluate effects of type of washer, weight of load, wash and rinse water temperatures, speed of agitation, duration of wash, speed of spinning, and various aspects of drying methods. For evaluation of some variables, Wrinklemeter and crease-recovery measurements were also used, and were themselves evaluated as measurement tools. S.B.D.

PATENTS

Degumming of Bast Fibres

Canadian Celanese *BP* 872,800 (U.S.A. 17 Apr 1959)

Decorticated bast fibre, particularly ramie, is treated with aqueous $KMnO_4$ at 85–100°C for 10–20 min and then with aqueous oxalic acid at $\leq$ 85°C until the pectin component of the fibre is dissolved and the hemicellulose component only slightly affected. Reducing the pectin component to $<$ 1% by weight of the finished fibre produces a free, non-self-adhering fibrous product which is not adversely affected by the residual hemicellulose. C.O.C.

Removing Graphite from Lace

Kayser-Bondor *BP* 873,201 (8 Oct 1957)

The lace is impregnated with a dispersion of casein, which is subsequently extracted from the lace. These steps are preferably alternately repeated over 15–60 min with about 60 yd/min of lace being treated. The dispersion is best extracted from the lace by passing it between squeeze rollers exerting a pressure of 10–80 lb/in^2. C.O.C.

Chlorite Bleaching of Cellulosic and Synthetic-polymer Fibres

H. Grunow and B. Mellbin *BP* 873,554 (France 11 Dec 1956)

Mono-, di- or tri-ethanolamine or their salts activate alkali- and alkaline-earth-metal chlorite solutions. The activated solutions are stable until heated, when a definite quantitative decomposition of chlorites occurs at $>$ 70°C. Thus cotton, spun rayon, or nylon is impregnated with 50–120% of its dry weight of an aqueous chlorite solution at pH 7·0–8·5 and 20–50°C. It is then heated to 70–100°C long enough to bring the duration of the entire process to 1–3 h. C.O.C.

Bleaching Acrylic Fibres and Films

Crylor *BP* 872,281 (France 7 July 1958)

The fibres or films are impregnated with a solution containing a chlorate and an acid compound capable of releasing chloric acid from the chlorate, either at room temperature or on heating. Finally they are steamed, preferably at 120°C. This yields an excellent bleach caused apparently by the joint action of the steam and chloric acid. C.O.C.

Improving the Efficiency of Dry-cleaning Solvents

Boehme Fettchemie *BP* 873,779 (Germany 17 Mar 1959)

Adding the mono- or di-saccharide of a fatty acid of $>$ 6 C much improves the detergent efficiency of organic solvents. Thus soiled wool and cotton, drummed in perchloroethylene at 25°C at a liquor ratio of 1 : 15 for 30 min, were much better cleansed when the solvent contained 1% on the wt. of the goods of sucrose monococonut fatty acid, and 0·1–0·5% of water. C.O.C.

Laboratory Methods for Measuring Washing Action (XIV p. 535)
Solvent Extraction of Leather (XIV p. 535)

VIII— DYEING

Dye Fixation in a Fluidised Bed. I— Synthetic Fibres

F. G. Audas *Dyer*, **125** (16 June 1961) 901–907 (30 June 1961) 937–940

This paper deals mainly with experimental work at the British Rayon Research Association on dyeing fabrics by a method involving padding, drying, and fixation of the dry dye at high temperatures, using a fluidised-bed machine. The first part deals with the machinery; disperse dyes on nylon, cellulose acetate, and Terylene, including a comparison between the fixation of insoluble (disperse) and soluble (Solacet) dyes of similar chemical type on Terylene; premetallised dyes on nylon and Acrilan. The second part deals with dispersed (unsulphonated) premetallised or metallisable dyes on nylon; modified basic (Sevron) dyes on acrylic fibres; azoic combinations on nylon, cellulose triacetate, and Terylene; and disperse reactiev (Procinyl) dyes on nylon. S.B.D.

Modifying the Dyeing Characteristics of Textile Fibres

E. Frieser *Spinner u. Weber*, **79** (Jan 1961) 14–17, 20–23 *Chem. Abs.*, **55** (12 June 1961) 11855

Methods are summarised for modifying cotton by esterification with acid chlorides, isocyanates, cyanuric chloride and benzyl chloride, by acetylation, by treatment with HCHO, and by aminising. Information is also given on modification of wool with tannin, phenol, sulphoamino acids and their derivatives, hydroxylamine salts, pyroracemic acid or urea, formaldehyde–naphthalenesulphonic acid condensates, polymethacrylic acid, phenylisocyanate, Mg bromoacetate, glyoxal and $NaHSO_3$, acetylation, acetylation with keten, and alkaline chlorination. Methods for nylon include treatment with monosulphonic acids, tannin and $SnCl_2$, thiophenols and their derivatives, inorganic salts, and condensates of naphthalene and its derivatives with HCHO or other compounds. Methods of making either component resistant to dyeing are given for the following mixtures:

wool with cellulose, polyamides or polyacrylonitriles; cellulose with polyacrylonitriles; polyacrylonitriles with polyesters; and cellulose acetates with cellulose. A table of resist agents is included. 37 References. C.O.C.

Studies on Direct Dyeing in Polymer Solution. III— Decrease of Apparent Affinity of Dyes for Cellulose and Heat of Adsorption for Polymer in Solution

Y. Suda and T. Shirota

J. Soc. Textile Cellulose Ind. Japan, 17 (May 1961) 414–420

The effects on the adsorption of dye by cellulose of adding wheat starch to the dyebath, and the influence of temperature, have been studied. The experiments were conducted with Chrysophenine G (C.I. 24895) and Direct Scarlet B (C.I. 22240). Addition of starch reduces the amount of dye adsorbed by the cellulose, and so results in a decrease in the apparent affinity of the dye. It is assumed that this is due to adsorption of dye by the starch (for which direct dyes have considerable affinity), thereby reducing the effective dyebath concn. The effective dyebath concn. $[D]_{\sigma'}$ and the difference between the apparent concentration $[D]_{\sigma}$ and $[D]_{\sigma'}$ (corresponding to the amount of dye taken up by the starch) have been calculated, and the following relationship has been shown to exist —

$$\frac{[D]_{\sigma} - [D]_{\sigma'}}{[D]_{\sigma'}} = K \text{ (constant)}$$

i.e., adsorption of dye by the starch obeys the distribution law. From the dependence of K on the temperature, heats of adsorption of dye by starch have been determined. The values obtained (12 kcal/mol for Direct Scarlet B and 11 kcal/mol for Chrysophenine G) are slightly lower than the corresponding heats of dyeing of cellulose (17 and 15 kcal/mol respectively). I.G.L.

New Developments in the Application of Cibacron Dyes

M. Peter *S.V.F. Fachorgan*, 16 (Apr 1961) 219–228

After discussing some of the properties of Cibacron (Ciba) reactive dyes (solubility, substantivity, diffusion, rate of fixation, and the stability of the dye–cellulose bond) the author describes the main dyeing methods by exhaustion (liquor circulation, jig, and winch machines) and padding. In the latter case there are six variants— pad–jig, pad–roll, cold pad–batch, pad–steam, pad–heat, and one-bath pad–steam. In all cases the unreacted dye should be removed by soaping aftertreatment. L.A.T.

Application of Cibacron Dyes by Cold Pad-Batch Method

P. Ulrich *S.V.F. Fachorgan*, 16 (Apr 1961) 241–247

In this method the padded fabric is batched into rolls and covered with plastic sheeting; gradual fixation of dye takes place overnight at room temp. The padding soln. is in a low state of aggregation, with little or no addition of urea and electrolyte. It is stable for about 1 h at 20°C. A high degree of chemical reaction between dye and substrate is achieved at dye concn. of 20–80 g/l. Other advantages claimed include— level and uniform dyeing, good colour yield, reproducibility of colour, ease of removal of unreacted dye, and absence of need for specialised machinery. L.A.T.

Infrared Fixation. A Further Possible Means of Fixation for Cibacron Dyes

I

P. Ulrich and W. Back

S.V.F. Fachorgan, 16 (May 1961) 298–313

The possibility of fixing reactive dyes by means of infrared radiation is discussed and compared with the Thermofix and Pad–Dry techniques. Two methods of infrared fixation are described— (1) two-stage fixation (intermediate drying) and (2) one-stage fixation. In method (1), cotton and viscose rayon were padded with identical mixtures and then fixed by either the infrared or the Thermofix process. For both dyes examined (Cibacron Blue 3G (C.I. Reactive Blue 2) and Cibacron Brilliant Orange GP (C.I. Reactive Orange 5) infrared fixation was far more rapid. For cotton, fixation took place within 10 s, and for viscose rayon within 20 s. Successful application to regenerated cellulose is a further advantage. Method (2) was compared to the Pad–Dry process. Infrared fixation is again rapid, being complete in 30–40 s for cotton (100 g/m²) and in 50–60 s for viscose rayon (135 g/m²). The amount of urea required was less than that needed in the Pad–Dry method. Comparative experiments were made with Cibacron Blue 3G and Cibacron Violet 2R (C.I. Reactive Violet 2); whereas a urea concn. of 200 g/l. was necessary for the Pad–Dry technique, for infrared fixation 100–200 g/l. was the most satisfactory concn. for application of Cibacron Blue 3G, and 50–100 g/l. for Cibacron Violet 2R. The greater the distance from the source of infrared radiation (lower fixation zone temperature) the longer was the fixation time; infrared fixation was more rapid than Pad–Dry fixation at all temperatures. Factors influencing the speed of reaction of a dye of this type with cellulose are discussed. A remarkable feature of infrared fixation is the lack of any dependence of the rate of fixation on the reactivity and substantivity of the dye. Fixation of all the monochlorotriazine dyes examined was found to take place within a few seconds. The fastness properties after infrared fixation are in no way inferior to those attained by alternative processes.

II

P. Ulrich and H. P. Schaub *Ibid.*, 314–319

Prints of Cibacron Black BG (C.I. Reactive Black 1), Cibacron Turquoise Blue GF (C.I. Reactive Blue 15), and Cibacron Brilliant Red B (C.I. Reactive Red 12) were made on cotton and viscose rayon. In each case three undried samples were subjected to infrared radiation (fixation zone temperature 150°C) for 20, 40, and 60 s respectively, and two samples, after drying, were fixed by the Thermofix process, again at 150°C, for periods of 1 and 5 min. Fixation of the cotton prints by the infrared method was found to be complete in 40 s and fixation of the rayon ones in 60 s. In terms of yield, infrared fixation was found to be slightly better on cotton, and far superior on viscose— the degree of fixation obtained by the Thermofix process proved to be insufficient. It is claimed that infrared fixation presents a means of combining the drying and fixing operations, with drying and fixation times far shorter, and urea requirements lower, than in alternative methods. Other advantages cited are higher reproducibility of the prints, elimination of any dependence on, e.g., steam fluctuations, and absence of any danger of bleeding during fixation. I.G.L.

Application of Drimarene Z Dyes by Padding

M. Capponi *S.V.F. Fachorgan*, 16 (Apr 1961) 229–236

Drimarene Z (Sandoz) reactive dyes can be applied by padding with or without intermediate drying. Glauber's salt inhibits dye migration during drying. The pad–roll and cold pad–batch methods do not require intermediate drying. In the former, dye fixation is carried out at 70–80°C, and in the latter at 20–25°C. L.A.T.

Yarn-dyeing with Phthalogen Dyes— I

G. Textor *Textil Praxis*, 16 (May 1961) 510–514

Details are given of the dyeing of cotton and cotton–staple-fibre yarns in cheese form with Phthalogen dyes. Recipes and process details are given. J.W.D.

New Development in Chemistry of Dyeing. III— Behaviour of Remazol Dyes when applied by Padding

H.-U. von der Eltz

Melliand Textilber., 42 (June 1961) 671–678

Part I (*ibid.*, 40 (1959) 69–73) dealt with the chemical basis of the Remazol dyes and their application, and II (*ibid.*, 40 (1959) 1443–1448) with factors involved in their dyeing by exhaustion. III is now concerned with the various factors of influence in padding. In the neutral bath these are affinity, migration during drying, and fixation conditions of alkali, temp., and duration. The advantages of the two-bath process are discussed. For the alkali-bath procedure, the effect of alkali on the stability of the padding liquor, fixation with $NaHCO_3$ or Na_2CO_3, and fixation with Na_3PO_4 and NaOH are discussed. These processes are compared. 15 References. H.H.H.

Recent Progress in the Application of Remazol Dyes

H.-U. von der Eltz and W. Hacker

Chemiefasern, 11 (June 1961) 404–409

Recent data are described which relate to the single-bath padding process, continuous dyeing procedures, and the

dyeing of the polyamide fibres Perlon, nylon, and Rilsan. Analogy with the application of acid dyes is noted. 10 References. H.H.H.

Continuous Dyeing of Woollen Tops
K. Fiedler *Textil Praxis*, **16** (Apr 1961) 383–385

The Irga–Pad (Geigy) process for continuous dyeing of wool tops with 1 : 2 metal-complex and milling acid dyes is described. The padding soln. contains the required amounts of dye, alginate thickener Lamitex L, wetting agent CH3288 (Geigy), formic acid, ethyl Cellosolve, and water. It is applied on a two-bowl padding mangle. Partial fixation of the dye takes place in a modified Fleissner steamer at just above 100°C for 90 s. Dye fixation is completed on passing the top through 0·5–4·0 ml/l. HCOOH (85%) at 60°C. The residue of thickener is removed by scouring. L.A.T.

Formation of Metal Complexes of Nitroso Dyes *in situ* on Protein Fibres
P. I. Mikhailenko

Trudy Khar'kov Politekh. Inst. im. V.I. Lenina, **26**, *Ser. Khim.-Tekhnol.*, (6) (1959) 113–121
Chem. Abs., **55** (26 June 1961) 12859

Wool, silk, and polyamide fibres were treated with 3% of a naphthol- or aminonaphthol-sulphonic acid at pH 3·2–4·0 at the boil for 30 min. They were then treated in the same bath after it is cooled with 3% $NaNO_2$ at pH 3–4 for 10–20 min at 3–5°C and then in a fresh bath with 1–2% of a metallic salt for 30 min at the boil. Among the acids used were 2-naphthol-6-sulphonic acid, chromotropic acid, Chicago 8S acid, and H acid. Addition of NaCl or Na_2SO_4 increased exhaustion of the acid, and presence of a wetting agent enabled the time of treatment to be reduced. Salts of Cr, Fe, Cu, Co, and Ni were used. When aminonaphtholsulphonic acids were used, the final product was a mixture of nitroso and azo dyes, and deep dyeings were obtained. The somewhat deactivated amino group of Chicago 8S acid was not diazotised under the conditions used, and an excellent deep colour resulted. If this amino group was acetylated, a different colour was obtained. Thus, use of Chicago 8S acid and acetylated Chicago 8S acid and various metallic salts enabled a full range of colours to be produced. The resultant dyes have better fastness than those obtained by the same dyes not formed on the fibre. When subjected to extraction with 25% aq. pyridine, nitroso dyes formed *in situ*, particularly the Fe and Co complexes, behave much like mordant dyes in that they are only partly extracted. This is because of lake formation: nitroso compound–metal–fibre. This was confirmed by forming similar complexes using gelatin and amino acids instead of wool. C.O.C.

Study in Union Dyeing
A.A.T.C.C. Northern New England Section

Amer. Dyestuff Rep., **50** (26 June 1961) P481–P486

Classes of dyes recommended for the fast dyeing of protein fibres, polyamides, modified acrylics, acrylic alloys, nytrils, and polyvinyl alcohols are reviewed, showing that the 2 : 1 metal-complex dyes have outstanding fastness properties together with simplicity of application. The problems involved in union dyeing of blends of any two of the above fibre classes are discussed for a one-bath method with 2 : 1 metal-complex dyes, and specific combinations are investigated. S.B.D.

Dyeing of Synthetic-polymer Fibres
A. A. Kharkharov and N. V. Ivanova

Zhur. Fiz. Khim., **35** (1961) 15–19
Chem. Abs., **55** (29 May 1961) 10897

Absorption maxima of some azobenzene dyes in polyamide, polyester, silk, and cellulose acetate fibres were all about the same (~ 426 mμ), indicating that the mechanism of interaction between each dye and the fibres is the same. The dyes used were 4-amino-, 4-(*NN*-dimethylamino)-, 4-(*NN*-diethylamino)-, 4-(*NN*-dimethylamino)-4′-methyl-, 4-(*NN*-dimethylamino)-4′-chloro-, 4 - (*NN* - diethylamino) - 3,5′- dichloro -, 4 - (*NN* - dimethylamino) - 4′- nitro -, 4 - (*NN* - diethylamino) - 4′- nitro-, and 4 - (*NN* - dimethylamino) - 3′- nitro - azo - benzene. Analysis of their spectra showed that they were bound to the fibre not by means of the π-electron interaction between dye and fibre but by salt formation. C.O.C.

Studies on the Dyeing of Acrylonitrile Fibre
Y. Takase

VII— Quantitative Analysis of the Nitrile Group in Polyacrylonitrile

J. Soc. Textile Cellulose Ind. Japan, **17** (May 1961) 421–424

With a view to establishing a suitable analytical method for the determination of the nitrile group in polyacrylonitrile, a number of representative acrylic fibres (copolymers and mixtures with other polymers, e.g. polyvinyl chloride) were analysed by the Kjeldahl method and by acid saponification. The Kjeldahl method gives reliable results in many cases, and its accuracy is improved if, prior to the Kjeldahl treatment, the samples are decomposed slowly with 1% hydrogen peroxide. This pretreatment can be used in conjunction with acid saponification for all types of acrylic polymers, including those containing nitrogen other than that in the nitrile group. Acid saponification takes place slowly, however, and may not be so satisfactory for copolymers and mixed polymers.

VIII— Various Methods of Preparing Cuprous Ions

Ibid., 424–427

Various methods of preparing cuprous ions from cupric sulphate solution, e.g. reduction of metals and organic compounds, etc., have been investigated and discussed in terms of redox potential theory. Irrespective of the method used, typical acid dyes will dye acrylonitrile fibre so long as cuprous ions are present, but it has been shown that no method is as satisfactory as that employing hydroxylamine sulphate when fuller depths are desired. For paler depths, however, alternative methods of cuprous-ion formation may find application. It has been found that the use of lactic or tartaric acid to adjust the pH (range 3–5) of ferrous sulphate soln. yields a soln. of low redox potential in which cuprous ions will be stabilised. I.G.L.

Dyeing Zefran Acrylic Fibre in Blends
F. M. Rawicz

Amer. Dyestuff Rep., **50** (29 May 1961) P415–P419

Zefran acrylic fibre can be dyed with many classes of dyes. The selection of these for the dyeing of this component in blends with wool, cellulosic fibres, or polyester fibres is discussed. S.B.D.

Saturation Limits of Disperse Dyes on Polyester Fibres
J. Carbonell *Textil Praxis*, **16** (May 1961) 501–510

Theory and practice of dyeing polyester fibres at high temp. are discussed. Detailed investigations are reported concerning the saturation limits of disperse dyes on polyester fibres at > 100°C, with special reference to the effect of duration of dyeing, type and quantity of dispersing agent, liquor ratio, influence of soaping, and effects of degree of exhaustion, fastness to rubbing, and uniformity of dyeing. Low concn. of dispersing agent leads to a high degree of exhaustion, better fastness to rubbing (especially after long dyeing times), but, with short dyeing times, unlevel dyeing. With liquor ratios ≮ 40 : 1 the concn. of dispersing agent must be calculated on the weight of fibre and not on the dye concn. For long dyeing times a very low concn. of dispersing agent is recommended. J.W.D.

Imperon MGN Process for Dyeing and Finishing Polyester Curtains
G. Weckler and S. Müller

Melliand Textilber., **42** (May 1961) 546–548

The dyeing and finishing of polyester net curtains is discussed in the light of recent developments and practical experience. Full details of the Imperon MGN process are given, and its advantages cited, e.g. low costs (small amounts of auxiliary products required), simple procedure, good colour reproducibility (even with different fixation temperatures), good fastness, and low tendency to soiling in use. Curtains treated in this way do not become grey, even on repeated washing. I.G.L.

Dyeing and Levelling of Polyester Materials
U. Lerch *Melliand Textilber.*, **42** (May 1961) 540–545
S.V.F. Fachorgan, **16** (June 1961) 403–414
Teinture et Apprêts, (63) (Apr 1961) 64–72

Levelling of disperse dyes on polyester fibres depends on the migration of the dye and not on rate of absorption.

Migration is influenced by—the carrier and its concentration in the dye bath; dyeing time and temperature; constitution of the dye. A carrier is necessary for dyeing at normal pressure to increase the rate of absorption and to make migration possible. The above requirements are shown to hold for dyeing at 100°C. For high-temperature dyeing, addition of 1–2 g/l. carrier is recommended. Choice of dyes is limited by the fastness properties demanded. There is, at present, no complete range with good levelling and at the same time good sublimation and pleating fastness. P.B.S.

Colouring Tinned Vegetables with Copper Compounds
J. W. Haken and G. M. Kastein
Conserva, **8** (1959/60) 54–56
Chem. Abs., **55** (12 June 1961) 11697

Spinach dyed with $\not> 20$ mg/kg Cu^{2+} has an unattractive colour; at > 80 mg/kg a homogeneous green is obtained which differs from that of freshly cooked spinach. At > 20 mg/kg formation of Cu chlorophyll occurs. At > 100 mg/kg an appreciable amount of free Cu^{2+} may appear in the product. Cu chlorophyll does not give level dyeing of tinned vegetables, but Na Cu chlorophyllin does and is effective at only 7–10 mg/kg (as Cu). C.O.C.

PATENTS

Preventing Migration during Pad Dyeing
ICI *BP* 871,193 (1 Apr 1959)

Cellulosic or man-made fibres are padded with a dispersion or solution of a non-cationic dye in an aqueous solution of an acrylic polymer containing both carboxy and carbonamido groups, a 0·1% aqueous solution of which has specific viscosity of 0·1–10·0 at 25°C. Presence of the acrylic polymer results in very level dyeings since it prevents the dye from migrating. C.O.C.

Stabilised Aqueous Solutions of Direct Dyes
General Aniline *USP* 2,955,011 (16 July 1956)

Presence of a little polyvinylpyrrolidone effectively prevents aqueous solutions of direct dyes from gelling or precipitation. The polyvinylpyrrolidone may be added to the dye before or after it is dissolved. Addition of polyvinylpyrrolidone to aqueous solutions of direct dyes which have thickened or gelled effectively restores the solutions to their original condition. C.O.C.

Dyeing Cellulose with Basic Dyes and Dyes containing Ternary or Quaternary Groups
ICI *USP* 2,955,900 (10 Jan 1957)

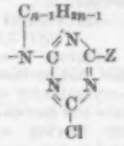
Affinity of cellulose, particularly of cotton and cuprammonium rayon, for the above dyes is greatly increased by treatment with polyacrylic acid and an alkali-metal acrylate. The dyeings are faster to wet- and dry-rubbing than those obtained without this treatment. Thus cotton is impregnated with its own weight of a 0·5% aqueous solution of a polyacrylic acid brought to pH 5·7 with NaOH, and dried at 100°C. It is then dyed at the boil in a bath containing the dye obtained by reacting Cu tri-(chloromethyl)-phthalocyanine with tetramethylthiourea, dyeing being carried out for 30 min while maintaining the pH at 3·5. It is then treated for 5 min in boiling 2% aq. Na_2CO_3 and soaped. It absorbs 1·25% of the dye, whereas an untreated sample absorbs only 0·6%. C.O.C.

Reduction of Vat Dyes
D. Goerrig *BP* 872,998 (Germany 2 Feb 1957)

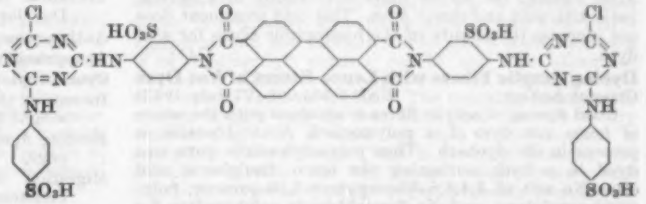
Vatting is accomplished more quickly than by any known method by reducing with an alkali-metal or alkaline-earth-metal borohydride in presence of a tetravalent S compound or a sulphonic acid. Thus Indigo (C.I. Vat Blue 1) (2 parts by wt.) is pasted with water (2 parts by vol.) and treated in water (20) with potassium borohydride (0·16). After 5 min, 30% NaOH (4 by vol.) is added and the bath is made up with cold water to 1000 parts by vol. Cotton is then dyed in the conventional manner in this dyebath at 20–25°C for 45 min. C.O.C.

Dyeing Cellulose with Reactive Vat Dyes
Ciba *BP* 872,949 (Switzerland 18 Apr 1957)

Cellulose fibres are dyed or printed from a bath containing an anthraquinone, perinone, or pericarboxylic acid imide vat dye containing at least one hydrophilic group and at least one group



(Z = Cl, subst. or unsubst. NH_2, unsubst. or etherified OH, $n \not> 5$), and converting to the leuco form by a reducing agent and effecting reaction with the fibre by an alkali. Cellulose is thus coloured in shades of excellent wet and light fastness and the process is simpler than conventional vat-dyeing techniques, being independent of temperature, and eliminating the vatting stage. Thus, to the dye (2)



in water (2000) are added Na_3PO_4 (20) and 20% NaCl (250), followed by cotton satin (100) at 20–25°C. The temperature is raised to 50–60°C over 20 min, and further 20% NaCl (250) added. After 30 min, Na hydrosulphite (5) is added and by dyeing at 50–60°C for 45 min a reddish violet dyeing is obtained which is oxidised by known methods, soaped, and rinsed to give a bluish red dyeing of good washing fastness. A.T.P.

Dyeing Protein Fibres with Reactive Dyes
ICI *BP* 874,513 (8 May 1959)

The fibres are treated in an aqueous dispersion of a water-insoluble reactive dye, i.e. one free from sulphonic and carboxylic acid groups and containing at least one group capable of reacting with the protein fibre, at pH < 7 and 80–100°C, and then the bath is brought to pH 4·5–8·0 and dyeing continued for a further period. Thus wool serge is dyed in an aqueous dispersion of 1,4-di-(γ-chloro-β-hydroxypropylamino)anthraquinone for 30 min at 100°C and pH 3·3, the bath is then brought to pH 7·5 and dyeing continued for a further 45 min at 100°C. This yields a bright blue dyeing of better fastness to wet treatments than a similar dyeing carried out throughout at pH 3·3.

USP 2,950,165 (10 Sept 1957)

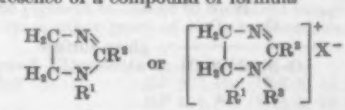
Dyeing is carried out at $\not< 55$°C in a bath containing a water-soluble azo or anthraquinone dye containing at least one *s*-triazinylamino group substituted in the triazine ring by $\not< 1$ Hal and an acid-binding agent. Alternatively the bath may contain either the dye at the start followed after some time by addition of the acid-binding agent, or *vice versa*. C.O.C.

Oxidation Dyeing of Human Hair
Wella *BP* 872,858 (Germany 21 Mar 1959)

Use of 3,4-diaminobenzoic acid or a salt thereof, together with an aromatic *p*-diamine and/or an aminophenol, yields bright, natural-looking light browns. C.O.C.

Level Dyeing of Filament Nylon Fabrics
Chemstrand Corpn. *USP* 2,952,506 (24 Aug 1956)

Level dyeings are obtained with direct, acid, premetallised acid, and afterchrome dyes if dyeing is carried out in presence of a compound of formula



(R^1 and R^3 = Alk, phenyl, hydroxyalkyl or carboxyalkyl; R^2 = aliphatic chain of 10–18 C; X = Hal or OH) e.g. 1,1 - di - (2 - hydroxyethyl) - 2 - heptadecylimidazolinium chloride. C.O.C.

Tone-in-Tone Effects on Cellulose Triacetate Fabrics
Celanese Corpn. of America
BP 872,544 (U.S.A. 20 Feb 1957)
USP 2,954,269 (20 Feb 1957)

Fabrics containing cellulose triacetate and no other thermoplastic fibres are hot-embossed, dyed, and then heated to increase the crystallinity of the cellulose acetate and completely eliminate the embossing. This results in a smooth-surfaced fabric having a tone-in-tone effect corresponding to the embossing received before dyeing. C.O.C.

Dyeing Acrylic Fibres with Acid and Direct Dyes
Chemstrand Corpn. *USP* 2,952,505 (14 Oct 1953)

Fibres containing < 80% of polyacrylonitrile and 2–10% of a poly-*N*-heterocyclic compound, if desired in blends with hydrophilic fibres, are treated for 10–30 min with a 0·01–1·0% aqueous acid solution at 160–212°F. After rinsing, the acrylic fibres dye readily in a neutral bath with acid and direct dyes. This acid treatment does not increase the affinity of the hydrophilic fibres for acid dyes. C.O.C.

Dyeing Acrylic Fibres with Leuco Esters of Vat Dyes
General Aniline *USP* 2,955,008 (27 July 1953)

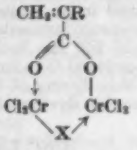
Good dyeing of acrylic fibres is obtained with the esters of leuco vat dyes if a polymerised *N*-vinyl-lactam is present in the dyebath. Thus polyacrylonitrile yarn was dyed in a bath containing the leuco disulphuric acid ester Na salt of 3,4,8,9-dibenzpyrene-5,10-quinone, polyvinylpyrrolidone, and Na formaldehyde sulphoxylate for 10 min at 140°F and 10 min at 190–200°F, sulphuric acid was added over 10 min and dyeing continued at the same temperature for 45 min. After oxidation and soaping, a golden yellow dyeing of excellent fastness to washing and very good fastness to light was obtained. C.O.C.

Dyeing Acrylic Fibres with Cationic Dyes
DuP *USP* 2,955,009 (28 Mar 1957)

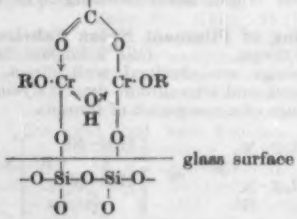
Level pastel dyeings and good exhaustion of the dyebath are obtained in presence of a non-substantive retarding agent, e.g. Na polystyrene sulphonate of mol.wt. 10,000–70,000. C.O.C.

Mordant Dyeing of Glass Fibres
J. P. Stevens & Co. *USP* 2,951,739 (20 May 1958)

The fibres, freed from carbonaceous matter, e.g. size, are treated with an acrylate chromic chloride of formula



(X = OH, H_2O or a monovalent anion of a monobasic acid; R = H or Alk of 1–7 C), e.g. one in which X = OH and R = CH_3. The strength of the solution used depends upon the depth of the desired dyeing. These compounds are not water-soluble and are first dissolved in isopropanol and then added to water. Conditions of treatment are 5–30 s at 70–100°F. The treated fibres are then heated to 225–375°F for 3–8 min. During this treatment volatile substances are driven off and the Cr reacts with the glass to yield



The fibres are then, with or without being washed, dyed with a mordant dye by entering into the dyebath at 120–160°F. After 30–300 s the bath is raised to 160–212°F and dyeing continued for 2–5 min. When the bath reaches 180°F, and preferably 200°F, a weak acid is added. This addition of acid shortens the time needed for dyeing and results in dyeings of better fastness. C.O.C.

Dyeing Leather, Skins, and/or Animal Fibres Attached Thereto
H. E. Nursten, L. Peters, and C. B. Stevens
BP 872,506 (9 May 1956)

The goods are treated with an organic compound which is not more than sparingly soluble in water at the temperature of dyeing and with an aqueous solution or dispersion of a dye, lake, or water-insoluble coloured organic compound. Thus chromed calf skin dyes more deeply with C.I. Acid Black 58 when n-butyl alcohol is present in the dyebath, and the wool of woolled sheepskin is dyed more strongly and brighter with Irgacet Scarlet RL when thymol is present in the dyebath. C.O.C.

Technical Aspects of Function, Drive and Control in Development of the Cyclotric Jig (I p. 503)
Anthraquinone Dyes for Polyolefins (IV p. 512)
Azopyrimidine Dyes for Polyacrylonitrile (IV p. 514)
Cyanoethylation of Cotton (VI p. 518)
Increasing the Affinity for Dyes of Linear, High-molecular-weight Poly-α-olefins (VI p. 519)
Binding Agents for Pigment Dyeing or Printing (IX this page)
Migration of Liquids in Textile Assemblies. IV— Penetration of Fabrics by Liquids (X p. 526)
Cross-linked Cotton or Rayon (X p. 528)
Ultrasonics— Applications to Dyeing in U.S.A. (XV p. 536)

IX— PRINTING

Emulsion-printing Processes
H. Wirth *Melliand Textilber.*, **42** (May 1961) 549–552

Various emulsion-printing processes are reviewed, e.g. Lupritol K (BASF), Saicatex (SAICA) and Savagraph (DuP) processes. Emulsion thickeners are divided into oil-in-water and water-in-oil types and their semi-emulsions. The advantages and disadvantages of these methods for use with various classes of dye are discussed. Emulsion printing of cellulose acetate or polyamides yields level prints after only a short steaming period, brilliance of hue and good levelling on acrylic fibres with no deterioration of handle and no need for pressure steaming, and brilliance of hue without the use of a carrier when applied to polyesters. I.G.L.

PATENTS

Binding Agents for Pigment Dyeing or Printing
FBy *BP* 872,526 (Germany 1 Dec 1956)

Dyeings and prints of excellent fastness to washing, ageing, and rubbing are obtained by using as binders aqueous emulsions containing a film-forming polymer plasticised with a polycondensate containing methylol groups obtained by condensing high-mol.-wt. *N*-methylol polyethers with low-mol.-wt. poly-*N*-methylol compounds. C.O.C.

Vigoureux Printing of Protein Fibres with Reactive Dyes
ICI *BP* 871,769 (8 May 1959)

Reactive dyes can be used in the Vigoureux printing of protein fibres if the printing paste contains, in addition to the usual thickeners, preferably Na alginate, and other assistants, an ethylene oxide–fatty alcohol or –phenol condensate. After the paste is printed on, the fibres are steamed at 100–104°C for 1–2 h, soaped, rinsed, and dried. C.O.C.

Printed Surface Covering and Printing Enamel therefor
Congoleum–Nairn *USP* 2,950,988 (9 Oct 1956)

A fibrous sheet is given a smooth decorative and wear-resistant coating < 0·0035 in. thick by printing it with a blend of solvent, pigments, resins, and a modified oil obtained by reacting a drying oil with an unsatd. carboxylic acid of 3–10 C and then esterifying it with a polyhydroxylic alcohol. C.O.C.

Photogravure Cylinders for Printing Uniformly Graduated Tinting Patterns on Plastic Sheeting
DuP *USP* 2,957,766 (27 June 1957)

Decoration of Synthetic Resin Reinforced with Glass Fibre or Fabric
C.I.M.E.M. dei Filli Gadani *BP* 871,751 (21 Apr 1958)

Glass fibre or fabric is printed and then coated with a resin precondensate and cured. This results in translucent sheets having a pattern of two or more colours. C.O.C.

Rendering Colourless Transfer Coatings containing Leucauramine Derivatives Fast to Light
Caribonum *BP* 873,702 (26 Aug 1958)

Many leucauramine derivatives, e.g. morpholinyl-leucauramine, on long exposure to light, tend to develop a yellow discoloration. This can be avoided by including *p*-benzylaminophenol, diphenylamine, *N*-phenyl-2-methyl-amine or their salts, or 1,5-dihydroxynaphthalene in the coating composition. C.O.C.

Transfer Material having a Magnetisable Coating
National Cash Register Co.
BP 867,933 (U.S.A. 8 Dec 1958)

A thin pliable base has one surface coated with a composition comprising particles of a magnetisable material, e.g. Black Magnetic Iron Oxide (C.I. Pigment Black 11), ferrite materials, cobalt–nickel alloys or powdered iron or steel (C.I. 77485), and as binder a linear dimethyl-siloxane polymer and a methyl and/or methyl-phenyl polysiloxane resin. It yields complete transfer of the coating onto a receiving surface over the whole area of applied marking pressure. C.O.C.

Magnetic Print Transfer Medium
General Electric Co. *BP* 873,696 (27 Mar 1958)

A magnetic transfer paper giving sharp and uniform impressions consists of a non-absorbent base sheet coated with a composition containing dispersed magnetic particles, e.g. Fe_3O_4 (C.I. 77499) (36%), castor oil (14), paraffin wax (10), and polyethylene (40). The base sheet must be a different material from the copy sheet, and adherence between the base sheet and the coating must be less than that of the coating for the copy sheet. C.O.C.

Water-slide Transfer
R. V. Wilks *BP* 873,613 (19 Dec 1956)

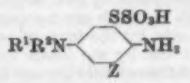
A paper sheet having a layer of water-soluble adhesive has a design printed on the exposed surface of the adhesive and this design is covered by a film of a strippable plastic. C.O.C.

Improving the Tonal Gradation of Diazotype Materials
General Aniline *BP* 871,216 (U.S.A. 10 July 1958)

A translucent or opaque base carries an upper layer containing a light-sensitive diazo component and a coupling component, and a lower layer containing the same or a similar light-sensitive component. The printing light is subject to the action of an ultraviolet absorber after passing the upper layer and before or during its action on the lower layer. The upper layer receives the shadow detail of the continuous tone image to be reprinted and the lower layer receives the highlights of the image. C.O.C.

Colour Photography
Ilford *BP* 872,683 (21 Jan 1959)

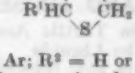
A silver salt image is developed in presence of a phenolic or amino colour coupler by a compound of formula



(R^1 and R^2 = same or different Alk or together with the N atom complete a ring system; Z = H, Alk or alkoxy) e.g. 4 - *NN* - diethylamino - 6 - methylaniline - 2 - thio-sulphonic acid. Depending on the colour coupler used, different types of image dyes are formed. When it is a phenol coupling preferentially *ortho* to the phenolic group, an oxazine dye results. When it is an amino compound coupling preferentially *ortho* to the amino group, an azine results. When it initially yields an indophenol or indamine dye, e.g. when 1-naphthylamine or 1-naphthol compounds are used, the dyes may be aftertreated with acids to yield thiazine dyes. Oxazine dye images produced by this method have excellent fastness to light. C.O.C.

Developers for Use in Reversal Process of Colour Photography
Kodak *BP* 873,198 (U.S.A. 3 Aug 1956)

Presence in the black-and-white developer of a compound of formula



(R^1 = H, Alk or Ar; R^2 = H or a salt-forming solubilising atom or group), e.g. 4-carboxy-2-(*p*-chlorophenyl)-thi-azolidine, reduces the tendency to fog. C.O.C.

Mordanted Photographic Imbibition Dye-printing Blank
Eastman Kodak Co. *USP* 2,952,566 (30 Oct 1956)

Imbibition dye-printing blanks are prepared by successively coating on a support, e.g. a baryta-coated paper, in any order, an aqueous solution of a hydrophilic organic colloid and thorium nitrate and a second solution of a hydrophilic organic colloid and Na aluminate sufficiently alkaline to precipitate metal hydroxide when the coatings meet each other. The resulting blanks yield sharper images and the hue shift from the wet to dry state of the print is much less than if the mordant is applied in known ways. This is probably because this method results in the dye image being formed much nearer to the surface of the paper, probably because the metal hydroxide is precipitated much nearer the surface or in a different form. C.O.C.

Photochemical Printing and Compounds therefor
National Cash Register Co.
BP 872,005 (U.S.A. 17 Sept 1957)

A base is coated with a photochromic compound which changes colour when exposed to light of an appropriate frequency only when the compound is in solution. It is then exposed to the required light in presence of an evaporable solvent for the compound printed in a pattern, the solvent then being evaporated to leave a fixed dye image. Suitable compounds include 1,3,3-trimethyl-indolino - 6′- nitrobenzopyrylospiran, 1,3,3 - trimethyl - indolino - 6′ - methoxy - 8′ - nitrobenzopyrylospiran, 1,3,3 - trimethylindolino - 6′- nitro - 8′- methoxybenzo - pyrylospiran, and 1,3,3 - trimethylindolino - 6′- bromo - 8′-nitrobenzopyrylospiran, which turn purple when exposed in solution to light of < 4500 Å, and 10-(9′,2′-methylxanthylidene)-anthrone, 3-methyl-10-(9′-xanthyl-idene) - anthrone, 3 - chloro - 10 - (9′- xanthylidene) - anthrone, and 10 - (9′- xanthylidene) - anthrone, which turn red under the same conditions, all these compounds being colourless before exposure. C.O.C.

Letterpress Printing on Plastics and Inks therefor
F. Lierg and O. Wolf *BP* 874,725 (Austria 29 Aug 1958)

Plastics are readily printed by the letterpress process if the ink used contains as binder a plastic of the same type as the plastic which is to be printed and which is dissolved in a solvent whose evaporation rate is 600 times less than that of ether. Thus polyvinyl chloride film is printed with an ink made up of a pigment and a solution of a vinyl chloride–vinyl acetate (87:13) copolymer in dibutyl oxalate. C.O.C.

Wet Xerographic Printing
Commonwealth of Australia
BP 873,080 (Australia 5 Apr 1957)

A photoconductor surface bearing an electrostatic latent image is immersed in a dispersion of a pigment in an insulating liquid. A biasing potential is applied through the liquid to produce a field in the opposite direction to the field induced by the xerographic image change. This causes the pigment to adhere to the latent image, but to be repelled from other parts of the photoconductor surface. C.O.C.

High-speed Electrostatic Printing
A. B. Dick Co. *BP* 871,194 (U.S.A. 10 May 1957)
BP 871,195 (U.S.A. 13 May 1957)

Colour Couplers (IV p. 510)
Aminopyridinediazonium Salts in Diazotype Printing (IV p. 512)

Dye Cyanides having a High Temperature-coefficient of Photochemical Reaction (IV p. 513)
Infrared Fixation. A Further Possible Means of Fixation for Cibacron Dyes (VIII p. 521)
Stabilised Aqueous Solutions of Direct Dyes (VIII p. 523)
Ornamenting Articles made from Polythene or Similar Plastics (XIII p. 532)

X—SIZING AND FINISHING

Migration of Liquids in Textile Assemblies. IV—Penetration of Fabrics by Liquids

F. W. Minor, A. M. Schwartz, L. C. Buckles, E. A. Wulkow, M. P. Marks, and G. H. Fielding

Text. Research J., **31** (June 1961) 525–539

Capillary penetration, which occurs solely as the result of capillary forces, and pressure penetration, caused by a slowly acting, external driving force, are similar in many respects. The major liquid property affecting these two types of penetration is the wicking power (which depends on the fabric and its finish as well as on the liquid). High wicking power results in rapid diminution of the surface density (wt. of liquid per unit superficial area of fabric wetted). At low surface density, penetration tends to be correspondingly low. Fabric tightness and denseness have the expected influence on capillary and pressure penetration, but the most important fabric property appears to be its fuzziness (quantity and stiffness of nap). The fabrics most resistant to penetration have a nap long and stiff enough to prevent contact between the substrate and the liquid-laden yarns. The nap fibres are shaped and spaced to resist being easily compressed to a wicking mass capable of conducting liquid to the substrate. Impact penetration is defined as the penetration resulting from a drop being thrown with appreciable momentum against a fabric. In this situation part of the liquid may penetrate instantly because of its momentum rather than being carried through the fabric by capillarity. Part of the liquid may also be dissipated by spattering. The most important factor in determining resistance to impact penetration appears to be the cover of the fabric. Fabrics having good cover, i.e. few or no straight-through channels from face to back, show the highest resistance. Appropriately constructed fabrics may resist penetration by a rapidly moving drop better than they resist capillary penetration by a drop of the same size. Test procedures and apparatus are described for measuring capillary, pressure, and impact penetration of fabrics by small, isolated masses of liquid. C.O.C.

Dirt-repelling Finishes

E. P. Frieser *S.V.F. Fachorgan*, **16** (June 1961) 382–391

Soiling of textiles is divided into dry, wet, and oily types. Only the first two types are considered, since the last is treated under oil-repelling finishes. Soiling depends upon fibre characteristics (cross-section, surface area, fat content, etc.) and on local conditions (airborne dust, relative humidity, etc.); various soiling mechanisms are listed. The ideal dirt-repelling finish is simple to apply and has little or no influence on dye or textile properties; available finishes are placed in five chemical classes and a short account of each class is given. 21 Textile finishes having dirt-repelling properties are compared by means of a soiling test and measurement of whiteness. P.B.S.

Influence of Material and Yarn Twist on the Raising of Cotton Flannels

E. Dworschak and R. Stoidl

Melliand Textilber., **42** (June 1961) 681–683

The lower the twist of the protective yarn, the greater the raisability and the more efficient the pile. Lower twist, however, gives reduced strength and stretch as compared with the higher-twisted yarn. Raising of the raw material produces a reduction in width which depends also on the twist, and appearance depends more on the nature of the material than on the twist. H.H.H.

Effect of Weft Yarn Twist on Properties of Cotton Fabrics Before and After Resin Treatment

E. Meshke

Amer. Dyestuff Rep., **50** (26 June 1961) P487–P493

Five samples of 24s cotton yarn, ranging from low- to high-twist, were spun and used for the weft in experimental fabrics of plain weave and average setts. The yarn with "average twist", highest strength, and least variability was used for two additional test fabrics— one of low sett and one of high sett. Untreated fabrics and those treated with ethylenene urea–formaldehyde resin were tested for crimp, stiffness, weight, thickness, tear strength, and crease recovery. All the fabrics showed considerable decrease in tear strength and increase in crease recovery after resin treatment. The fabric made with the weft yarn of highest strength with average sett had the highest tear strength and also the best crease recovery after resin treatment. Handle and appearance ratings tended to favour the fabrics of lowest weft-yarn twist. Interpretations of test results indicate the importance of fabric geometry and the influence of yarn twist upon the properties of resin-treated cottons. S.B.D.

Effects of Tension during Resin Treatment on Physical Properties of Cotton Fibres and Yarns

R. S. Orr, A. W. Burgis, and J. N. Grant

Text. Research J., **31** (June 1961) 550–557

Changes in strength and elongation caused by resin treatment of yarns result from changes in strength and elongation of the fibres. The loss of strength resulting from treatment in absence of tension is caused principally by cross-linking of the fibres in positions unfavourable for equal bearing of the load on the fibre. Although application of even low tension during treatment tends to reduce the loss in strength, there is further loss in elongation. Energy to rupture of yarn treated under tension is only slightly greater than that of yarn treated in absence of tension. Use of high-strength, high-elongation cottons and mercerising before the resin treatment partly offset the low toughness achieved for the average cotton. Application of tension before drying, followed by low tension during curing, also minimises the loss of toughness in yarns. C.O.C.

Formamide–Formaldehyde Products as Crease-resist Agents for Cotton Fabrics

S. L. Vail, J. G. Frick, P. J. Murphy, and J. D. Reid

Amer. Dyestuff Rep., **50** (12 June 1961) 437–440

The properties of cotton fabrics treated with formamide–formaldehyde products are described and it is concluded that the finish produces wash-and-wear fabrics, but that the finished fabrics are susceptible to damage from hypochlorite bleaching. The mechanism is believed to involve cross-links of both formaldehyde and formamide–formaldehyde types. W.G.C.

Infrared Condensation of the Dimethylolurea–Catalyst Mixture

W. Vauch *Melliand Textilber.*, **42** (June 1961) 690–696

The course of this condensation is followed to obtain practical data for plant requirements and working conditions. A theoretical treatment is included, and data for various inorg. catalysts are recorded, from which it appears that the ammonium salts (e.g. NH_4Cl, $(NH_4)_2SO_4$, $NH_4H_2PO_4$, and NH_4CNS) play a special role. Optimum results are found to depend on the type of infrared radiator and the amount of inorg. catalyst used. H.H.H.

Chlorine Retention of Resin-finished Materials

H. A. Wannow

S.V.F. Fachorgan, **16** (June 1961) 392–402

Textiles which have been treated with nitrogenous resins or reagents react with bleaching agents containing chlorine to form chloramine compounds. This combined chlorine leads to fibre damage on heating. The extent of this damage is determined by the "scorch test" methods of the AATCC and SNV, which are compared. Fibre damage is influenced not only by the type of resin and conditions of application, but also by the fabric itself. Differences in test results with different fabrics are found to be due to retention of alkali, which leads to increased chlorine retention. This can be prevented by an acid treatment before application of the resin. P.B.S.

Problems of Storage of Crease-resistant Cotton

P. Moruzzi *Tinctoria*, **58** (May 1961) 185–188

Normal and accelerated (at 80°C) ageing tests have been used to investigate the evolution of formaldehyde and the retention of crease resistance of five formaldehyde finishes. Washing the cloth in a non-ionic detergent improves the life of the finish. Although urea–formaldehyde resins decompose slightly more rapidly than the others, the finished cloth retains its crease-resistant characteristics better. E.V.T.

Comparison of Ultraviolet Radiation Absorbers for Protecting Wool against Yellowing
W. G. Rose, M. K. Walden, and J. E. Moore
Text. Research J., **31** (June 1961) 495–503

Substituted benzophenones effectively protect wool against yellowing by ultraviolet (UV) radiation. The protection given depends on factors other than simple absorption of UV radiation, because compounds having similar UV absorption characteristics vary greatly in the protection they afford. In addition, the method of application of a given UV absorber strongly influences the amount of protection secured. Water-soluble UV absorbers applied as though they were dyes give the best protection, nearly complete protection being obtained by use of 2,4-dihydroxybenzophenone-2′-ammonium sulphonate in hot aqueous solution at pH 1·3–1·7. C.O.C.

Application Principles of Acrylic Backcoating
C. E. Hoey
Amer. Dyestuff Rep., **50** (29 May 1961) P410–P414

For many years fabrics have been backcoated to upgrade their end-use characteristics. Added body and ravel resistance were among the first improvements obtained. Chemical improvements in the binding agents have greatly enhanced their durability to wet processing. With the ever-increasing trend to highly styled fabrics containing long floating designs, a major portion of the strength burden is placed on the resin binder. The superior ageing characteristics of acrylic resins on exposure to heat or sunlight, or to both, and their ease of compounding are the principal reasons for their acceptance for various backcoating operations. The use of backcoating is discussed for upholstery fabrics, carpeting, and apparel. S.B.D.

PATENTS

Applying Wax or other Lubricants to Yarn During Winding
Leesona Holt *BP* 870,628 (2 Oct 1957)

The capillary properties of a porous metal are used to provide a film of molten wax or liquid lubricant on the surface of the metal while yarn is passing over it. The yarn is uniformly treated irrespective of any variation in room temperature. Visual and/or audible warning is given when the supply of wax falls below a predetermined level. C.O.C.

Permanent Crimping of Cellulosic Yarns—K-set Process
American Viscose Corpn. *USP* 2,950,521 (22 May 1957)

Cellulosic yarn is crimped, e.g. by knitting it into tubing, and is then given a thermoplastic finish, e.g. water-soluble cellulose ether (1–6%), aldehyde (1–10), acidic cross-linking catalyst (2·3) and, if desired, lubricant (0·5) and a resin (1–10), cured and then unravelled. C.O.C.

Catalyst for Aminoplast Finishes
Rohm & Haas Co. *USP* 2,950,553 (16 Jan 1957)

Use of Mg chloride or perchlorate, or Zn nitrate or perchlorate or fluoroborate as catalyst enables curing of aminoplast condensates to be effected so rapidly that mangling or hand-ironing suffices. Thus fabric containing cotton is impregnated with an aminoplast precondensate and one of the above catalysts and dried at < 200°F. It is then made into a garment and pressed at $\not< 212$°F to set the aminoplast. Forming the garment before setting the aminoplast removes the difficulties encountered in sewing resin-finished fabrics and in forming permanent pleats or creases in them. C.O.C.

Resin Finish having no tendency to retain Chlorine
Joseph Bancroft & Sons Co.
USP 2,952,892 (19 July 1956)

Use of a mixture of a polyacrylic ester and a silicone resin gives good crease-recovery properties and water-absorbency, a good handle, high abrasion resistance, and no chlorine retention. C.O.C.

Modification of Cellulosic Textiles
ICI *BP* 869,659 (31 Dec 1957)

Cotton or rayon is treated with a compound containing no chromophor, at least one 1,3,5-triazine ring substituted on C by at least one Hal, SO_3H, or SO_2X (X = alkali metal or alkaline-earth metal) at least one primary or secondary amino group attached by its N atom, and at least one salt-forming solubilising group. It is then treated with an agent capable of reacting with the groups introduced into the cellulose by the first treatment. Thus cotton cloth treated with Na 2-(4,6-dichloro-*s*-triazin-2-yl)-aminonaphthalene-6-sulphonate and Na_2CO_3, dried at 110°C, soaped, and then treated with $CuSO_4$, cetyl pyridinium bromide, or $CoCl_2$ has good resistance to attack by micro-organisms. Alternatively, if the second treatment is with Na stearate then a soft handle which is fast to hot soaping results.

BP 869,660 (15 Jan 1958)

Cellulose is treated with a compound containing at least one 1,3,5-triazine ring substituted on C atoms by 2 Hal, a primary or secondary amino group bound by its N atom, and at least one positively charged or non-ionic solubilising group. Thus cotton cloth treated with an alkaline solution of *p*-(4,6-dichloro-1,3,5-triazin-2-yl) aminophenacylmethylammonium chloride, dried at 110°C and treated at the boil with a non-ionic detergent, had good fastness to attack by micro-organisms. Similar treatment with β-(4,6-dichloro-1,3,5-triazin-2-ylamino)-ethanol imparted a soil-resistant finish to cotton and a soft handle fast to washing on rayon. C.O.C.

Treating Native Cellulose with a Liquid Alkylene-polyamine and a Resin
U.S. Secretary of Agriculture
USP 2,955,014 (4 Mar 1955)

Native cellulose fibres are treated with a solution containing 55–95% by wt. of liquid alkylene polyamides and 45–5% of inert liquids until an alkylenepolyamine–cellulose complex is formed. The unchanged alkylene-polyamines are then removed. This reduces the crystallinity of the fibres and permanently swells them. The treated fibres have a wool-like handle and have a greater affinity for dyes than the untreated fibres and are more readily penetrated by impregnating agents. Resin treatment of such fibres results in much less loss of strength than with the untreated fibres. C.O.C.

Incorporating Thermosetting Resins in Linen Fabrics
Tootal Broadhurst Lee Co. *BP* 871,105 (19 Apr 1956)

A better smooth-drying finish than has hitherto been available for linen fabrics is obtained by using in both stages of the process of *BP* 437,361 (*J.S.D.C.*, **52** (1936) 194) a low-viscosity precondensate of 1·4–2·5 mol. of urea and 1 mol. of HCHO. In the first stage the fabric must absorb 1–13% of the precondensate and in the second stage 1–9% the total taken up in the two stages being 4–18%. C.O.C.

Preventing Acetone–Formaldehyde Resin Finishes from Discolouring
Calico Printers' Association *BP* 872,150 (17 Mar 1958)

The tendency of goods finished with acetone–formaldehyde resins to become discoloured is removed by washing them in a solution containing an oxidising agent. C.O.C.

Flame-resistant Cellulose Derivatives
Textile Research Institute
BP 873,555 (U.S.A. 31 Dec 1956)

Cellulose is esterified in the 6-positions of its glucose anhydride unit. Then at least some of the ester groups are reacted with aqueous metallic halides to introduce halogen into the cellulose molecule and yield a flame-resistant product. Thus cotton cloth was mercerised in absence of tension, washed free from alkali, soured with acetic acid, and washed free from acid. It was then partly dried at 50°C and impregnated with pyridine at room temperature and immediately treated in a bath of methanesulphonyl chloride in pyridine at 65°C for 5–10 min. Finally it was rinsed, soaped, and dried. The product did not have sufficient flame-resistance for many purposes, so it was treated with an aqueous metal halide at pH 7 to replace the OSO_2CH_3 group by halogen, excellent flame-resistance being obtained by use of iodine and good by use of bromine.

BP 873,556 (U.S.A. 31 Dec 1956)

The fibres are treated with a dialkylhalogenophosphate in pyridine at < 100°C, preferably < 40°C, for < 60 min, successively or simultaneously with the esterification. The product should contain 0·2–2·0% by weight of phosphorus, this being sufficient to prevent afterglow when the fibres are burned or charred. Treatment with an aqueous metal halide will increase the flame resistance. C.O.C.

Cross-linked Cotton or Rayon
Rohm & Haas Co. *BP* 873,492 (U.S.A. 29 Jan 1957)

Fibrous products, > 50% by wt. of whose fibres are cellulosic, are cross-linked by treatment with a salt of an amine of formula $R_{3-n}N(\cdot AXCH{:}CH_2)_n$ (n = 2 or 3; R = cyclohexyl, benzyl, or Alk of 1–8 C; A = alkylene of 2–4 C and provides a bridge of < 2 C between X and the N atoms; X = O or S). The treatment also confers affinity for acid dyes and markedly improves the affinity for direct and other dyes. Thus cotton cloth is impregnated with methyl-di(2-vinyloxyethyl)amine dissolved in aqueous formic acid, dried at 240°F, and cured at 325°F. The treated fabric has good crease-recovery properties and dyes much deeper with direct dyes than does the untreated cloth. C.O.C.

Smooth Fabrics from Cotton and Crimped Yarns of Synthetic Polymer
Heberlein & Co. *BP* 873,282 (Austria 17 Jan 1959)

Smooth fabrics are obtained from cotton yarns and crimped yarns of synthetic polymers by treating the fabric made from them with mercerising lye until the cotton is fully shrunk. C.O.C.

Diazotype Reproduction Cloth
Winterbottom Book Cloth Co. *BP* 873,325 (4 Apr 1959)

A calendered starch-filled cloth is coated with a water-resistant resin and next with a hydrophilic colloid. The cloth is then ready to receive a diazotype layer applied from aqueous solution. If an abrasive, e.g. Aerosil, is present in the colloid layer, it renders the final cloth receptive to pencil markings. C.O.C.

Setting of Keratinous Fibres
Unilever *BP* 872,828 (5 Feb 1959)

When keratinous fibres are set, e.g. by crabbing, steaming or use of thioglycollates, improved results are obtained if they are esterified before the disulphide bonds are broken. This results in a set obtained by steaming being fast to washing or to production of sharper or tighter pleats or creases. C.O.C.

Reducing the Felting Properties of Wool
Precision Processes (Textiles) *BP* 872,292 (28 June 1956)

The wool is treated with an aqueous solution of permonosulphuric acid or salts thereof and a permanganate at pH < 11. Treatment with H_2SO_3 or a sulphite removes the brown stain and also further reduces the felting properties of the wool. C.O.C.

Reducing the Felting Properties of Animal Fibres
N. Corah (St. Margaret) *BP* 874,630 (26 June 1959)

The goods are treated with sodium chlorite and sodium hypochlorite, preferably by adding these agents in that order to the bath at pH 4–8 in which the goods are already immersed. C.O.C.

Modifying Keratins by Treatment with Sulphones and Related Compounds
U.S. Secretary of Agriculture *USP* 2,955,016 (15 Oct 1956)

Organic compounds, containing at least two olefinic unsaturated groups linked to a sulphone, sulphoxide, or phosphine oxide radical are excellent cross-linking agents for use with reduced keratins. They may be applied after or simultaneously with the reducing agent. Thus solvent-extracted white flannel was soaked for 1 h at 50°C in 0·35-M aqueous 2-mercaptoethanol (30 ml/g of wool) and rinsed with water. It was then treated for 30 min at room temperature in divinyl sulphone in 0·1-M Na borate (2 millimole sulphone in 50 ml of the solution per g of wool), rinsed, and dried. The alkali- and peracetic acid-solubility of the treated wool and its supercontraction were much less than those of the untreated wool. C.O.C.

Modification of Nylon
DuP *BP* 872,514 (U.S.A. 2 July 1956)

Nylon is treated with an acidic compound containing aliphatic unsaturation and is then heated to polymerise the compound without altering the chain length of the nylon. This imparts an antistatic finish, increases the m.p., and confers soil-resistance. The modified nylon may be hot-embossed and has increased crease-recovery powers in that fabrics becoming creased in use are smoothed out by merely wetting and hanging up to dry. Thus nylon fabric treated with aqueous acrylic acid containing a little NH_4 persulphate for 30 min, heated at 90°C for 1 h, and then washed 6 times for 1 h each time in distilled water at 60°C, is given an antistatic finish and higher resistance to hole-melting. C.O.C.

Grafted Condensation Polymers having Good Antistatic and Soil-resistant Properties
DuP *BP* 872,513 (U.S.A. 2 July 1956)

Antistatic and soil-resistant properties are given to condensation polymers by grafting into them structures containing acid or salt groups (other than carboxyl or carboxylates) derived from reactive compounds containing non-aromatic C–C unsaturation. The grafting may be effected by high-energy particulate or electromagnetic ionising radiation. Thus nylon is impregnated with an aqueous solution of K styrene sulphonate and then irradiated with a dose of 20 Mrep. It is then rinsed with methanol to remove excess of monomer, and then for 30 min in acetone to remove surface polymer. After repeated washings it has better antistatic, soil, and hole-melting resistance than untreated nylon. C.O.C.

Flame-resistant Finish for Hydrophobic Fibres
American Cyanamid Co. *USP* 2,953,480 (18 Nov 1953)

Impregnating the fabrics with an aqueous solution of NH_4Br and an aminotriazine–aldehyde precondensate and then curing imparts a good flame-resistant finish to fabrics of nylon, cellulose acetate, acrylic fibres, etc. The finish is not hygroscopic, does not tend to dust off on repeated flexing, and is fast to dry cleaning. When exposed to flame, the charred beads formed have negligible tendency to separate and drop off the fabric and there is no afterglow. C.O.C.

Permanent Finish on Acrylic Fibres
Crylor *BP* 870,701 (France 2 Feb 1959)

The fibres are impregnated with a latent solvent containing a polymer which is soluble, at least on heating, in the solvent. Impregnation is effected at a temperature below that at which the fibres dissolve in the solvent, and the impregnated fibres are dried at or above the temperature of dissolution. Thus polyacrylonitrile fabric is immersed for 30 min in a 2% solution of polyacrylonitrile in dimethylformamide at 20°C. Excess of liquor is centrifuged off, leaving 28% in the fabric, which is then dried for 2 h at 60°C and then for 30 min at 120°C. This gives a light-weight permanent finish without affecting the strength of the fabric. C.O.C.

Impregnating Woven Fabric with Polyvinyl Chloride
N. J. Brown *USP* 2,955,958 (5 Mar 1956)

The fabric is passed for 2–5 s through a solution of polyvinyl chloride and a plasticiser in a volatile solvent at 120–140°F and is then dried at 140–160°F. This yields a waterproof and crush- and crease-resistant fabric having a soft handle, and good draping properties, and which is readily tailored. Because the polymer does not penetrate the fibres there is no loss in tear or impact strength. C.O.C.

Stretchable Fabric having a Suède or Velvety Finish
American Viscose Corpn. *USP* 2,951,005 (24 Dec 1953)

A reticulated, fibrous web of elastomeric fibres, either alone or bonded to an elastic or stretchable woven or knitted fabric, is coated with flock, e.g. by rendering the elastomeric fibres tacky by treating with a solvent or an adhesive before applying the flock. This flocked coating enables the product to slide readily over the skin, e.g. when used in bathing costumes or corsets, and also permits decorative effects to be obtained. C.O.C.

Felting and Tip Weathering of Wool (VI p. 519)
Changes in Density of Steelon Filament in Relation to Draw Ratio and Heat Treatment (VI p. 519)
Cause, Effect, and Prevention of Pilling (VI p. 519)
Imperon MGN Process for Dyeing and Finishing Polyester Curtains (VIII p. 522)
Printed Surface Covering and Printing Enamel therefor (IX p. 524)
Coated Webs and Laminates (XI p. 529)
Aminoplasts (XIII p. 531)
Acrylic Coatings Cross-linked with Amino Resins (XIII p. 531)
Silicones in Industrial Finishes (XIII p. 532)
New Types of Linoleum (XIII p. 532)
Improving the Fastness to Light of Laminates containing Aminoplasts (XIII p. 532)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Synthetic Latices as Binders for the Clay Coating of Paper

H. Yasuda and V. Stannett *Tappi*, **44** (Feb 1961) 96–102

All-latex acrylate copolymer (I) is stronger than all-latex styrene–butadiene (II), but the position is reversed with starch–latex blends. Polyvinyl acetate homopolymer (III) is poor, but vinyl acetate–dibutyl maleate copolymer (IV) shows promise. Varnish hold-out is best for I with all-latex, but II is better with starch blends. I, II, and IV give excellent gloss and II is outstanding for smoothness, while brightness and opacity are hardly affected. Starch blends give stiffer coatings than all-latex. R.A.

Storing and Preservation Conditions of Wet Spruce Mechanical Pulp

II—Ion-exchange Properties of Spruce Groundwood Pulp with Special Regard to the Fungicide Phenyl Mercury Acetate (PMA)

K.-E. Eriksson, H. Kiessling, and B. Steenberg

Svensk Papperstidning, **64** (15 May 1961) 329–335

In addition to physical absorption of PMA, phenylmercuric ions are bound to the carboxylic groups of the pulp by ionic bonds. They cannot be displaced by metallic ions, but are displaced by quaternary ammonium ions. Halogen ions decrease the retention effect. R.A.

III—Mechanism of Inactivation of PMA by Groundwood Pulp

K.-E. Eriksson *Ibid.*, **64** (31 May 1961) 381–385 (*in English*)

Mercury can migrate from the tertiary wall in the wood cells where it is primarily retained, through the cell walls towards the middle lamella, if the impregnated wood is stored in de-ionised water at 80°C. Breakdown of PMA is probably due to aldehydic groups in the wood. R.A.

PATENTS

Fortified Rosin Size

American Cyanamid Co. *USP* 2,957,777 (31 Oct 1957)

Paper size consisting essentially of the partly or completely neutralised Diels–Alder reaction product of rosin with 2–15% (on free and combined rosin present) of acetylene dicarboxylic acid is much more effective, weight for weight, in imparting water-resistance than is an ordinary rosin size. C.O.C.

Paper Manufacture

Hercules Powder Co. *BP* 871,205 (U.S.A. 31 Mar 1958)

Porosity and absorbency are improved, without loss of uniformity of formation, by using cellulose cross-linked with formaldehyde or a compound having $\not< 2$ functional groups reactive with cellulose. R.A.

Fluorocarbon-sized Paper

Minnesota Mining and Manufacturing Co. *USP* 2,920,979 (13 Jan 1956)

Carbon, wrapping, and similar papers are made by sizing a porous, unsized paper on one side with a Cr complex of a fluorocarbon monocarboxylic acid and coating the other side with a hot-melt wax or asphalt composition. R.A.

Currency Paper

U.S. Secretary of Commerce *USP* 2,920,984 (30 Mar 1953)

The elasticity and crumpling resistance are improved by immersion in a bath containing 15–20% by wt. of glycerol, mannitol, or sorbitol, 10–15% urea–formaldehyde resin, and the remainder water, and drying at $\not< 60$°C. R.A.

Coloured Board or Paper

Diamond National Corpn. *USP* 2,920,991 (14 Jan 1954)

The stock is beater-coloured and the web formed from it coloured at the calender stack with an aqueous solution of a different hue, after which a coloured mineral-adhesive composition is imprinted on the surface. The method allows easy control of uniformity of colour. R.A.

Paper-machine Coating

Beloit Iron Works *USP* 2,920,698 (12 Oct 1955)

The Yankee dryer drums are covered with a heat-resistant hydrophobic material, e.g. Teflon. To ensure easy release, the web is removed from the drums, except the last one, while only partially dried. R.A.

Coated Paper

O. J. Meijer's Fabrieken voor Verpakte Artikeln *BP* 871,937 (Holland 11 Feb 1958)

The paper is coated with an aqueous composition containing starch and/or a starch derivative, part of which is non-gelatinised and/or non-dissolved, and then immediately brought to above the temperature at which the starch and/or its derivative is dissolved, the rise in temperature being so rapid that all the starch and/or its derivative is gelatinised or dissolved before the water in the composition and in the paper has completely evaporated. This enables cold-swelling starches to be used, the compositions are either thixotropic or have low viscosity and good flowing properties with fairly high solid content, and the finished paper has high gloss, good pick resistance, and excellent printing properties. C.O.C.

Coating Regenerated Cellulose Film with Polyethylene

DuP *USP* 2,953,482 (18 July 1957)

A little of an organic peroxide is added to polythene dissolved in a volatile organic solvent and the solution immediately applied to the film. The coated film is then dried below the decomposition temperature of the peroxide and then further heated to decompose the peroxide and cure the coating. This yields a firmly adherent, printable coating. C.O.C.

Coated Webs and Laminates

Unilever *BP* 869,994 (Germany 31 July 1957)

A non-thermoplastic web is united to another layer by means of an intermediate layer formed by the hot-roller fusion of thermoplastic particles. C.O.C.

Graft Polymerisation of Some Acrylic Esters with Cellulose Substrates

American Cyanamid Co. *USP* 2,955,015 (5 June 1957)

A cellulose substrate is graft-polymerised with ethyl, butyl, or propyl acrylate in an aqueous medium at pH $>$ 3·5 and in presence of a ceric salt soluble in at least one component of the reaction mixture. Thus paper treated with an aqueous emulsion of butyl acrylate in absence of oxygen but in presence of a ceric solution (0·1-M $Ce(NH_4)_2(NO_3)_6$, 1-N HNO_3) for 60 min at 22·5°C, picked up 5 times its weight of butyl acrylate to yield a product having very flexible and soft qualities, very much like chamois leather. C.O.C.

Pressure Cooking for Coating Colour Preparation (III p. 504)

Improving the Fastness to Light of Laminates containing Aminoplasts (XIII p. 532)

Factors Affecting the Determination of Carboxyl Groups in Cellulose (XIV p. 535)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Tanning Studies with Dialdehyde Starch—Preliminary Evaluation of the Leather

E. M. Filachione, I. D. Clarke, E. H. Harris, J. Fee, L. P. Witnauer, and J. Naghski

J. Amer. Leather Chem. Assocn., **56** (May 1961) 200–212

Acetone-dehydrated calfskin was tanned with dialdehyde starch under various conditions: dialdehyde starches of three levels of oxidation (33, 67, and 96%), two pH levels (8 and 10), two methods of dissolving the starch, and with the leather either oiled or unoiled. For this tannage, optimum leather properties were obtained with the 96%-oxidised starch, at pH 10, and with the leather oiled. The highest area yield was obtained with the 67%-oxidised starch, added as solid to the tanning liquor, at pH 10, and with oiling of the leather. Optimum weight-yield was obtained with the two starches of low oxidation level, at pH 10, after oiling. J.W.D.

Developments in Combination Tannages. III—Stability of Chrome Retan Leather Under Warm Humid Conditions

D. A. Williams-Wynn and R. L. Sykes

J. Soc. Leather Trades Chem., **45** (May 1961) 214–220

Ageing at 45°C and $>$ 95% r.h. for three weeks is shown, with chrome retan leather, to correspond to ca. 9 months natural ageing when little or no organic acid is present. Correlation is not obtained with leathers containing appreciable quantities of organic acids, or with those that have been retanned with blends of syntans and

vegetable extracts. Deterioration appears to be the result of hydrolysis under acid conditions, and moisture plays a vital role; when moisture is excluded there is much less deterioration. J.W.D.

Kinetics of Chrome Tannage
J. C. de Wijs *J. Soc. Leather Trades Chem.*, 45 (June 1961) 242–250

The course of Cr uptake by hide powder with time can be expressed, as a first approximation, by the function $(Cr_2O_3) = Kt^b$, where (Cr_2O_3) is the amount of Cr_2O_3 bound, t is the tanning time, and K and b are constants. When the expression is used to show the influence of the basicity of a Cr liquor on the uptake of Cr, these constants are related to the mean electric charge of all cationic Cr complexes in the tanning liquor. J.W.D.

PATENT

Cleaning Plastics and Leather and Decolorising Leather
H. C. Turner *BP* 874,720 (5 May 1959)

The surface of the plastic or leather is treated with a stream of air containing particles of a hard organic and/or inorganic material other than sand. The goods are cleaned at a low pressure with particles just light enough to clean the surface and are then treated with an air pressure and particle size regulated to produce the desired finish. Leather so cleansed and decolorised is in a very suitable state for re-dyeing. C.O.C.

Structure of Fibres of the Collagen Group and Related Matters (VI p. 518)

XIII—RUBBER; RESINS; PLASTICS

Newer Ways of Making Polymers—A Guide for the Non-Chemist
M. Kaufman *J. Plastics Inst.*, 29 (June 1961) J59–J63

Polymers and polymerisation are simply defined and the historical development of polymers is outlined. Addition polymerisation, stereospecific polymerisation, and block and graft copolymerisation are described in simple terms and illustrated. W.R.M.

Modern Aspects of Polymers
J. J. P. Staudinger *Chem. and Ind.*, No. 23 (10 June 1961) 771–776

All the newer developments in plastics extend the temp. range within which they can be used without impairment of properties. This is directly, though not exclusively, connected with their ability to crystallise, which in turn has its basis in the structure of the molecule. From this standpoint polyolefins, polydienes, olefin copolymer rubbers, polyoxymethylene, and polycarbonates are discussed. J.W.D.

Irradiation of Polymeric Materials
R. M. Black *Trans. Plastics Inst.*, 29 (June 1961) 98–105

The effects of irradiation of polymers with high-energy radiation are briefly discussed. The predominant chemical effects are cross-linking and chain scission, and the effects of these on physical properties are outlined. The possible use of polymers in insulation for nuclear environments is discussed and radiation processing is briefly considered. Some applications of irradiation of polyethylene, polypropylene, natural rubber, silicones, and the formation of graft copolymers are outlined. W.R.M.

Deeply Coloured, Electrically Conducting Polymers
I—Preparation and Properties
P. V. French, L. Roubinek, and A. Wassermann *J.C.S.*, (May 1961) 1953–1963

Cyclopentadiene, cyclopentenyl chloride, cyclopentadienylidenecyclopentane, and a colourless cyclopentadiene polymer are converted, under catalysis by trichloroacetic acid in aprotic solvents (e.g. benzene and CCl_4), into unsatd. polymers of mol.wt. 1090–30000, which are deeply coloured (like aq. 0·1-N iodine), owing to the tail end of light-absorption peaks at 310–365 mμ, and accept protons reversibly. The electronic spectra of the protonated polymers are characterised by peaks between 470 and 650 mμ. These polymers are proved to be highly unsatd., containing a longer and a shorter sequence of conjugated double bonds.

II—Mechanism of Formation
J. Murphy, L. Roubinek, and A. Wassermann *Ibid.*, 1964–1975

The stoicheiometry of polymerisation, which is formally, but not kinetically, a polycondensation, the degree and rate of polymerisation, and oxidative degradation are discussed. Of general interest is a method of calculus used for the solution of a differential rate equation relating to two consecutive reactions. H.H.H.

Diffusion in Glassy Polymers. I—Effects of Initial Concentration upon the Sorption of Organic Vapours in Polymers
H. Odani, S. Kida, M. Kurata, and M. Tamura *Bull. Chem. Soc. Japan*, 34 (Apr 1961) 571–576 (*in English*)

In glassy polymer–organic vapour systems, the sorption kinetics exhibit three types of non-Fickian anomaly (sigmoid, pseudo-Fickian, and two-stage) as the initial concentration for each differential step increases, and the differential absorption process for amorphous systems changes to the normal Fickian type at the penetrant concentration at which the given polymer–vapour mixture undergoes glass transition. This study is concerned with integral absorption experiments in which the initial concentrations were taken in regions corresponding to these four types of differential absorption process. The systems studied were atactic polystyrene + benzene and cellulose nitrate + acetone—both at 25°C. In the former case successive differential absorptions changed with initial concentration according to the scheme—sigmoid→ pseudo–Fickian → two-stage → Fickian. The rate of sorption of benzene is strongly dependent on the benzene concentration when this is low (0·095 g/g), but is less so with increase in the concentration of benzene, and the rate is almost independent of benzene concentration when the critical concentration is exceeded. The cellulose nitrate–acetone system was not studied so exhaustively. The successive differential absorption curves for this system are—sigmoid → pseudo–Fickian → two-stage type → sigmoid. In the glassy state the dependence of integral absorption rates upon the penetrant concentration does not differ much from that for the atactic polystyrene–benzene system. I.G.L.

Structure and Properties of Isotactic Polyacrylic Acid and its Salts
V. A. Kargin, S. Ya Mirlina, V. A. Kabanov, G. A. Mikheleva, and A. V. Vlasov *Doklady Akad. Nauk S.S.S.R.*, 135, No. 4 (1 Dec 1960) 893–895

Isotactic polyacrylic acid was prepared by alkaline hydrolysis of isotactic polyisopropyl acrylate. The completion of hydrolysis was controlled by potentiometric titration or titration to phenolphthalein. The isotactic acid was precipitated from the aq. soln. with HCl. It is a white crystalline, hydrated powder containing 1 mol. H_2O for every 2 monomeric chains. From electron microscopic examination it appears that the acid soln. exists in the form of amorphous globules or their aggregates, depending on the concentration. It has a slight tendency to crystallise in presence of HCl. This is connected with the complete lack of dissociation of –COOH groups and the regularity of its chains. Its Ba salt assumes the form of compact, dense globules and has lower viscosity than the acid. The Na salt also assumes the form of globules at pH < 6·75. The globules disperse at pH 6·75 owing to the increase in dissociation of COOH and an electrostatic intramolecular repulsion. At pH 7·5 dissociation is complete. The chains assume chemical regularity; this promotes an internal irregular crystallisation and the formation of highly orientated regions. At a higher pH the excess of molecular ions screens the repelling charge of the macromolecules, which decreases the internal electrostatic repellency. The chains assume the form of electrostatic balls. The properties of isotactic sodium polyacrylate are enhanced in the tetramethylammonium salt. T.Z.W.

Polycondensation on the Phase Boundary
A. P. Tereht'ev, I. G. Mochalina, and E. G. Rukhadze *Doklady Akad. Nauk S.S.S.R.*, 138 (No. 5) (11 June 1961) 1130–1131

The synthesis of a polyamide with average DP of 20,000–30,000, using the interphase polycondensation

method, is described. A benzene solution of the dichloro-anhydride of pyridine-2,6-dicarboxylic acid is reacted with an aq. sol. of hexamethylenediamine. The polyamide is formed either as a film on the boundary between liquid phases or in the form of powder when stirring is applied.
I.G.

Adsorption of Iodine by Nylon 6
S. Pacetti *Tinctoria*, **58** (Apr 1961) 148–152

The adsorption isotherms at 20, 30, and 40°C of iodine (from I_2 in aqueous KI) on films of nylon 6 have been investigated. The characteristics of the isotherms, the phenomena of reversibility and hysteresis, the rate of desorption in a stream of moist air, and the X-ray diagrams indicate that there is a temperature-independent critical concn. of iodine in the polyamide at which remarkable changes in the physical properties of the amide are evident. The critical concn. represents the accessibility of the polyamide to iodine. For nylon 6 recrystallised by treatment with boiling water for 18 h, the accessibility is 0·507 g/g, and for non-recryst. nylon 6 it is 0·576 g/g.
E.V.T.

Pyrolysis of Linear Copolymers of Ethylene and Propylene
S. Straus and L. A. Wall
J. Res. Nat. Bur. Stand. A, **65** (May–June 1961) 221–226

Plots of rate of volatilisation against percentage volatilisation for linear polymers and copolymers of ethylene and propylene show maxima, suggesting some random degradation. The maximum rate increases with increasing amount of propylene in copolymers. Minute amounts of impurities in polypropylene lower the rate of volatilisation, and it is suggested that this may be due to inhibition of a free-radical process. The activation energy for decomposition of a fully deuterated linear polyethylene is very close to that for a non-deuterated one. Similar studies on a β-irradiated linear polyethylene suggest that the radiation causes chain scission as well as cross-linking. W.R.M.

Assignment of the Infrared Absorption Bands and the Measurement of Tacticity in Polypropylene
M. P. McDonald and I. M. Ward
Chem. and Ind., No. 19 (13 May 1961) 631

The infrared spectra of isotactic and atactic polypropylene, isotactic poly(2*d*-propylene), and isotactic poly(3,3,3d_3-propylene) have been examined; all the bands in the polypropylene spectra have been assigned, together with most of the analogous vibrations in the deuterated polymers. A monomer unit is defined as being in an isotactic state when both its adjacent neighbours on the chain are identically substituted sterically to itself. If the chain configuration is helical this monomer unit will then show certain specific molecular vibrations. On this view the infrared spectrum cannot distinguish either crystallinity or tacticity *per se*, but only a chain configurational state. J.W.D.

Diffusion in Glassy Polymers. II—Effects of Polymer–Penetrant Interaction; Diffusion of Ethyl Methyl Ketone in Atactic Polystyrene
H. Odani, J. Hayashi, and M. Tamura
Bull. Chem. Soc. Japan, **34** (June 1961) 817–821 (*in English*)

The successive differential absorptions for the atactic polystyrene (A-PS)–ethyl methyl ketone system change with the initial concn. according to the scheme: sigmoid type → pseudo-Fickian type → two-stage type → pseudo-Fickian type → Fickian type, the last transition occurring at lower concn. than that for benzene (cf. Tamura *et al.*, *ibid.*, 571 and *J. Phys. Chem.*, **64** (1960) 594). Moreover, the second-stage portion of the two-stage process shifts rapidly to the short-time regions as the initial concn. of each step increases, at a rate similar to that in the (A-PS)–benzene system (*loc. cit.*). The general features of integral absorptions for both systems are very similar irrespective of the difference in the penetrant–polymer interactions.
H.H.H.

Infrared Transmission and Emission by Polytetrafluorethylene (PTFE)
T. Wentink and W. G. Planet
J. Opt. Soc. Amer., **51** (June 1961) 601–602

The infrared absorption spectrum of PTFE at 298°K from 2–15 μ and the surface emission spectrum at a minimum temperature of 700°K are reported. The apparatus is described in the preceding paper by the same authors (p. 598), where similar results for quartz are discussed. The materials studied were heated by an arc wind tunnel, and the radiant energy was focussed to the slits of a modified Perkin–Elmer model 83 prism spectrometer. Strong absorption occurs at < 3 μ, owing to optical scattering in the opaque, crystalline polymer, which becomes optically clear at > 324°C. The only significant absorption in thin sections (0·025 mm) out to 6 μ is that due to a C–F vibrational overtone at 4·2 μ. This is in agreement with the observed emission peak at 4·2 μ. The emissivity varies with wavelength in accordance with absorption, so that assignment of a colour temperature and description of the hot surface as a grey-body are not valid.
E.COATES

Aminoplasts
J. Brzezinski and Z. Wirpaza
Monograph, 422 pp., 201 figs., 65 tables, publ. Warsaw, 1960
Polish Tech. Abs., No. 3/4 (39–40) (1960) 161–162

The first monograph to be published on the subject in Poland covers the development, production, and application of amino resins, including theoretical considerations, synthesis, properties, and analysis, together with information on world production and the uses in various industries. R.A.

Symposium on Thermosetting Acrylic Resins
H. L. Gerhart *Off. Dig. Fed. Soc. Paint Technol.*, **33** (June 1961) 680–683

Introduction to the Symposium, outlining the areas of recent development and improvement of resins of this type. J.W.D.

Thermosetting Compositions from the Reaction of Acrylamide Interpolymers with Formaldehyde
R. M. Christenson and D. P. Hart
Off. Dig. Fed. Soc. Paint Technol., **33** (June 1961) 684–698

Solution interpolymers of acrylamide and several other monomers, having relatively low mol.wt., have been subsequently reacted with HCHO in the presence of alcohols to form methylol or etherified methylol substituents on the pendant amide groups. These polymer soln. may be applied by spraying and heat-cured to form insol. films of high performance characteristics. Acrylates, methacrylates, styrene, and vinyltoluene are particularly useful monomers in the first stage. The polymers are readily compatible and coreactive with numerous other resins, both natural and synthetic, increasing the scope of their usefulness. J.W.D.

Acrylic Coatings Cross-linked with Amino Resins
J. C. Petropoulos, C. Frazier, and L. E. Cadwell
Off. Dig. Fed. Soc. Paint Technol., **33** (June 1961) 719–736

Thermosetting systems prepared by reacting acrylic copolymers containing varying amounts of acrylamide, methylolacrylamide, β-hydroxyethyl methacrylate (I), or methacrylic acid with aminoformaldehyde resins, are compared with conventional industrial baking finishes. Copolymers containing I have the best overall film properties. An attempt is made to correlate properties with chemical composition and structure. J.W.D.

Epoxy Resins in Thermosetting Acrylics
D. D. Applegath *Off. Dig. Fed. Soc. Paint Technol.*, **33** (June 1961) 737–746

Many acrylic interpolymers can be thermoset through epoxide curing mechanisms, by use of many types of epoxy resin. Aliphatic liquid epoxy resins may be used where the more viscous phenolic epoxides are unsuitable owing to excessive solvent requirements or cured film brittleness. It may be possible to adapt a given acrylic resin to several dissimilar uses simply by blending it with different epoxy resins prior to curing. J.W.D.

Hexahydrophthalic Anhydride in Alkyd Resins
S. E. Berger and A. J. Kane
Off. Dig. Fed. Soc. Paint Technol., **33** (May 1961) 597–606

Alkyd resins formulated with *cis*-1,2-cyclohexanedicarboxylic (or hexahydrophthalic) anhydride yield soln. having low viscosity at high solids content. Coatings based on these resins have excellent flexibility, high abrasion resistance, light colour, and good colour retention. Careful

formulation, particularly with regard to the choice of drier (and its concn.), leads also to coatings of satisfactory hardness and drying characteristics. J.W.D.

Silicones in Industrial Finishes
H. L. Cahn *Off. Dig. Fed. Soc. Paint Technol.*, 33 (May 1961) 607–621

High-temp.-resistant silicone industrial finishes can be produced in a variety of vehicles and in any colour required. Several applications of industrial finishes of this type are briefly discussed. Ten illustrations. J.W.D.

New Types of Linoleum
I. D. Kozlovskiĭ and F. K. Kompaniets
Prom. Ekon. Byull. Sovet. Narod. Khoz. Lugansk. Ekon. Admin. Raĭona, (4) (1958) 28–30
Referat. Zhur. Khim., (1959) Abstr. No. 76743
Chem. Abs., **55** (29 May 1961) 10916

Improvement of the properties (especially resistance to light and frost) and lowering of the cost of linoleum were achieved by using finely dispersed pyrite cinders (the waste product from H_2SO_4 production). This improved the plasticity of the linoleum because of the 2·3% of S present and imparted a fast light brown to dark red, depending on the amount of cinders present (10–25%). Simultaneously, almost complete replacement of deficient H_3BO_3 by Al_2O_3, and of tricresyl phosphate by triphenyl phosphate was accomplished while the cellulose ester content decreased from 28 to 15% and in some cases to 10%. The resulting linoleum had high water-absorption capacity compared with standard linoleum because $CaSO_4 \cdot \frac{1}{2}H_2O$ was used instead of $CaSO_4 \cdot 2H_2O$. Replacement of tricresyl phosphate by triphenyl phosphate reduced costs by up to 20%. The linoleum type NLP so obtained is 3·5 times cheaper than the standard linoleum type NL. C.O.C.

PATENTS

Incorporating Carbon Black (C.I. Pigment Black 6 and 7) in Polymer or Copolymers of Ethylene and Propylene
J. Zomlefer *USP* 2,952,656 (16 July 1957)

Homogeneous, stable, non-phasing mixtures of large amounts of Carbon Black with polyethylene, polypropylene, or ethylene–propylene copolymers useful as moulding or extrusion powders yielding strong flexible products are obtained by adding the pigment to a thin solution of the polymer at a temperature about its gel point and while constantly stirring. The hot dispersion is cooled, stirring being continued until a gel is formed, after which the solvent is removed. The product is friable and readily broken into moulding powder. C.O.C.

Extrudable Polyethylene Pigmented with Carbon Black (C.I. Pigment Black 6 and 7)
Union Carbide Corpn. *USP* 2,951,821 (23 Jan 1956)

Tough solid polyethylene pigmented with a 1·5–5·0% of a mixture of two Carbon Blacks, the mixture having a mean volume–surface diameter of 32–44 mμ, has a reduced tendency to adsorb moisture compared with compositions containing Carbon Blacks of smaller particle size and so consistently possesses better durability. Because of its physical and light absorption properties it is more suitable for outdoor use than similar compositions containing Carbon Blacks of larger particle size. C.O.C.

Ornamenting Articles made from Polythene or Similar Plastics
W. Baulcombe and A. C. Cartwright *BP* 874,960 (7 Jan 1958)

A stencil has dyed or pigmented paper applied to one side of it and then that side is pressed against the plastic article. Pressure and heat are then applied through the cut-out portions of the stencil by means of a hot platen having relief portions corresponding precisely with the cut-out portions of the stencil. C.O.C.

Aqueous Dispersions of Nylon
General Dispersions *USP* 2,951,054 (16 Nov 1955)

Nylon is dissolved in a hot mixture of alcohol and water, the temperature being so controlled that neither the alcohol nor the water distils off. The solution is then diluted to a content of 3–6% nylon by weight and is fed slowly into a dispersing zone containing a boiling mixture of alcohol and water. The resulting dispersion is then passed to a still in which the alcohol is removed to leave an aqueous dispersion of nylon. C.O.C.

Aromatic Linear Polyester Solutions containing Dyes or other Additives
FBy *BP* 873,591 (Germany 24 July 1958)

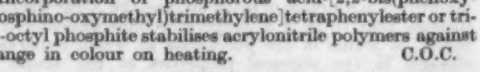
Anhydrous hydrofluoric acid is an excellent solvent for polyesters, 20–50% solutions being readily obtained at < 20°C. Dyes, e.g. vat or phthalocyanine dyes, can also be readily dissolved at the same time as the polyesters, or delustring or cross-linking agents or plasticisers may also be added. C.O.C.

Stabilisers for Acrylonitrile Copolymers
American Cyanamid Co. *USP* 2,951,055 (30 Jan 1959)

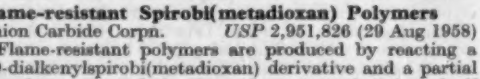
Incorporation of phosphorous acid-[2,2-bis(phenoxyphosphino-oxymethyl)trimethylene]tetraphenylester or tri-iso-octyl phosphite stabilises acrylonitrile polymers against change in colour on heating. C.O.C.

Flame-resistant Spirobi(metadioxan) Polymers
Union Carbide Corpn. *USP* 2,951,826 (29 Aug 1958)

Flame-resistant polymers are produced by reacting a 3,9-dialkenylspirobi(metadioxan) derivative and a partial phosphate ester derivative of pentaerythritol and, if desired, simultaneously with an aliphatic polyhydric alcohol. Thus 3,9-divinylspirobi(metadioxan)



(160 g)



(100 g)

and octylphenol acid phosphate (1) were heated for 4 h at 100–130°C. The mixture was then cooled to 45°C and mixed alkane sulphonic acid (1·3) added. Finally it was poured into moulds and cured for 16 h at 100°C. This yielded a hard and tough polymer having a very light colour and which was self-extinguishing, even when ignited repeatedly by the method described in ASTM–D635–44. C.O.C.

Self-sterilising Plastics
Steril-plast *BP* 871,228 (9 Sept 1959)

Incorporation of 2,2′-thio-bis-(4,6-dichlorophenol)-2,2′-methylene-bis-(3,4,6-trichlorophenol) or a mixture of the two, renders plastics self-sterilising, even after repeated washings. C.O.C.

Decorative Laminates and Surface Coating Composition therefor
General Electric Co. *BP* 874,326 (U.S.A. 18 Apr 1957)

Laminates consisting of a core layer supporting a pattern layer and a wear layer of resin-impregnated translucent overlay paper can be satisfactorily prepared by using instead of the overlay paper a finely divided cellulosic material of refractive index equal to that of the paper. If this is done it enables a silica-containing coating composition to be used which not only improves the abrasion resistance of the product but also improves the clarity and definition of the pattern. C.O.C.

Improving the Fastness to Light of Laminates containing Aminoplasts
Ciba *BP* 874,179 (Switzerland 16 May 1957)

2,4-Dihydroxybenzoylfuran gives excellent fastness to light to aminoplasts and to colorants in or beneath the aminoplasts. C.O.C.

Improving the Fastness of Organic Polymers to Ultraviolet Radiation
DuP *BP* 872,421 (U.S.A. 1 Aug 1957)

An ultraviolet absorber compatible with the polymer and of lower m.p. is applied to at least one surface of the polymer, which is then heated to between the m.p. of the absorber and that of the polymer until some of the absorber has penetrated to a limited depth in the polymer, e.g. so that most is just below the surface and the amount decreases to zero within 20% of the thickness of the polymer. There should be at least 0·4 g/m² of absorber. C.O.C.

Light Stabiliser for Halogeno Resins (III p. 506)
Pigments and Dyes for Plastics (IV p. 507)
Effect of γ- and Neutron-radiation on the Properties of a Coat of Varnish (V p. 517)
Epoxy-modified, Water-soluble Alkyd Resins becoming Hydrophobic when Cured (V p. 517)

Increasing the Affinity for Dyes of Linear, High-molecular-weight Poly-α-olefins (VI p. 519)
Readily Dyeable Polyolefins (VI p. 519)
Coloured Polyamides (VI p. 519)
Printed Surface Covering and Printing Enamel therefor (IX p. 524)
Synthetic Latices as Binders for the Clay Coating of Paper (XI p. 529)
Graft Polymerisation of Some Acrylic Esters with Cellulose Substrates (XI p. 529)
Cleaning Plastics and Leather and Decolorising Leather (XII p. 530)

XIV— ANALYSIS; TESTING; APPARATUS

Physical Methods in Organic Chemistry

A. R. Pinder

I— Practical Techniques

Chem. and Ind., No. 23 (10 June 1961) 758–770

A review, with 36 references, embracing: adsorption, paper, partition, electrophoretic, and gas chromatography; zone refining; countercurrent distribution; organic electrode processes (polarography, cathodic reduction, anodic oxidation, and anodic substitution); colorimetry; and mass spectrometry. Other techniques are briefly referred to. J.W.D.

II— Physicochemical Measurements

Ibid., No. 31 (5 Aug 1961) 1180–1192

An account of the methods used to measure the physical properties of organic cpd. from which information concerning mol. structure and configuration can be derived. Each section gives a brief description of apparatus, a theoretical discussion of principles involved, and examples of problems to which the particular method has been applied. Spectroscopy is divided into sections dealing with U.V. and visible absorption, Raman, I.R., microwave, nuclear magnetic, and electron spin resonance spectroscopy, with examples taken from several fields of organic chemistry. Other subjects include X-ray crystal analysis, with a brief description of electron-density projection maps, and electron diffraction and its application to the stereochemistry of 1,2-dichloroethane. Optical rotation is discussed under mol. rotation differences and optical rotatory dispersion, with a description of the Cotton effect. Application of dipole moments and dissociation constants to the study of geometrical isomers is mentioned. 37 Group references, mainly to text-books and reviews, are given. F.J.

Acid-Base Equilibria. I— Calculation of Titration Curves

J. P. Escarfail and W. Simon

Helv. chim. Acta, **44** (15 June 1961) 977–985

An equation for the calculation of titration curves of arbitrary mixtures of acids and bases is derived and applied numerically, using a digital computer. Examples are given for four acids (HCl, β-bromopropionic acid, *p*-nitrophenol, and *p*-methoxyphenol) with a strong base (NaOH), of four bases (NaOH, ethanolamine, triethanolamine, and quinoline) with a strong acid (HCl), of β-bromopropionic acid with ethanolamine, of ethanolamine with *p*-nitrophenol, and of a mixture of acids with a strong base and of bases with a strong acid. H.H.H.

Estimation of Concentration of Surface-active Agents with *p*-Toluidine

K. D. Podshibyakina

Tekstil. prom., No. 3 (Mar 1961) 63–64

p-Toluidine hydrochloride forms complex salts with sodium salts of alkylarylsulphonic acids. On addition of caustic soda, a reverse reaction occurs giving *p*-toluidine and sodium salts of sulphonic acids. This property has been utilised to develop a method for estimation of alkylaryl sulphonates. The detailed procedure is given. L.S.L.

Determination of Hydroxy and Amino Compounds Using Pyromellitic Dianhydride

S. Siggia, J. G. Hanna, and R. Culmo

Anal. Chem., **33** (June 1961) 900–901

Definite advantages over acetic and phthalic anhydrides are claimed for the title compound in the determination of alcohols and amines. Its reaction rate with alcohols is comparable to that of acetic anhydride, and aldehydes do not interfere. Because it does not react with phenols, it also provides a rapid method for determining alcohols in the presence of phenols. P.B.S.

Resin - stabilised 4 - Trimethylammoniumbenzenediazonium Cation as a Reagent for Amines and Hydroxy Compounds

J. L. Lambert and F. Zitomer

Anal. Chem., **33** (June 1961) 960–961

Diazonium cations can be stored on dry nuclear sulphonic ion-exchange resins for long periods (up to 2 years) and released into solution when needed by exchange with an inorganic cation. The title compound is claimed to be superior to previously described reagents. Details of its preparation and use are given, with a short table of the colours of the azo dyes formed with typical compounds. P.B.S.

Microscopic Methods in Practical Investigations and Research

S. Hummel *Textil Praxis*, **16** (Apr 1961) 398–402

After reviewing briefly the main methods of microscopy the author describes in some detail, and illustrates with photomicrographs, specific microscopic techniques which enabled diagnosis and differentiation between apparent and real cases of "milky" stripiness in continuous-filament viscose rayon. The former was caused by a residue of very small particles of inorganic matter, e.g. sulphur, and the latter by organic substances, or gas bubbles trapped in hollow cavities between the core and skin of the filament. L.A.T.

Two Attachments for the G.E. Spectrophotometer

A. Brockes and O. Koch

Die Farbe, **9** (Dec 1960) 267–272

Two attachments for the G.E. (General Electric) Hardy Spectrophotometer are described. A reflectance attachment with 45° incidence and normal detection is outlined, in which a photomultiplier tube of the type RCA 6217 in a small integrating sphere is used to give high sensitivity. The radiation beams from the monochromator are focused by means of lens systems to a smaller sample area than normal and finally pass into the detector sphere through iris diaphragms, which allow of 100% line setting. Another attachment is described in which a xenon lamp is situated in an integrating sphere to give diffuse illumination on normal sized samples (5 × 4 cm), from which radiation is collected at 6° down the monochromator system. This serves for the measurement of reflectance factors of fluorescent samples. E.Coates

Some Observations on the Colour Measurement of Fluorescent Materials

A. Berger and O. Koch *Die Farbe*, **9** (Dec 1960) 259–266

The principles involved in the measurement of the reflectance of fluorescent materials are indicated in comparison with the standard requirements for measurements on non-fluorescent bodies. The necessity for a source of radiation with an ultraviolet content is stressed, and the use of the xenon arc is mentioned. Spectrophotometric systems require the dispersion of the reflected radiation when fluorescent materials are under study, as distinct from most normal systems, in which the selected radiation is reflected immediately prior to detection. Some commercially available instruments are considered as to suitability and accuracy in such measurements. E.Coates

The Concepts and Specification of Colour Rendering by Light Sources

W. Münch *Die Farbe*, **9** (Dec 1960) 143–182

The physics and the physiology of the colour of light sources and bodies under irradiation are discussed. The important terms involved in colour rendering are interpreted and the methods of measuring and specifying colour rendering are outlined. Some practical applications of the methods, particularly on problems of lighting, are considered. E.Coates

An Inquiry into Colour Rendering by Light Sources and its Specification

I. Hennicke *Die Farbe*, **9** (Dec 1960) 183–206

Various methods (visual means, test colour computation, and spectral band methods) are compared for assessing the colour-rendering properties of six warm-white lamps. All of these methods give essentially the same order of ranking to the lamps. In the visual method the result

obtained for seven carefully selected test colours was the same as for forty-four colours. The current formulae used for the evaluation of colour differences in the test colour computation method all yielded practically the same order for the lamps. The same was true for the Spectral Band Indexes. E.COATES

Specification of Colour-rendering Properties of Fluorescent Lamps
J. L. Ouweltjes
Die Farbe, **9** (Dec 1960) 207–246 (*in English*)

The comparison of commercial fluorescent lamps has been made in general between normal and de luxe types, where there are pronounced differences. This work describes a method for computing the spectral-energy distributions (SED) of fluorescent lamps from those of available phosphors and combinations thereof. Some fifty SED curves for each of the three main lamp colours (white, warm-white, and daylight) were obtained. The test-colour method of specifying colour rendering is discussed and it is shown that eight suitably chosen colours are adequate for the purpose. The Adams formulae can be considered useful for calculating colour differences. Different formulae (Nickerson–Stultz and Saunderson–Milner) give slightly different results, but the influence on lamp rating is negligible. The application of the spectral band method (SBM) to the rating of lamps is considered and the shortcomings of the CIE Band System are discussed. No entirely satisfactory correlation of the SBM with the test-colour method was found, and the latter is considered to be the more reliable method. E.COATES

Polarometric Titration of Some Azo Acid Dyes with Benzidine
M. Matrka and Z. Ságner
Chem. průmysl., **10** (1960) 638–639
Chem. Abs., **55** (26 June 1961) 12857

A method based on formation of insoluble salts of azo acid dyes and benzidine has been applied to dyes containing two SO_3H groups, and particularly to Crystal Ponceau 6R (C.I. 16250) and Brilliant Yellow (C.I. 24890). Soln. (55 ml) containing $1{\cdot}25 \times 10^{-4}$ moles of dye and 2·5-N HCl (6 ml) was titrated under N_2 with 0·025-M benzidine in 0·125-N HCl. A mercury dropping electrode was the cathode and satd. Hg_2Cl_2 the anode. The results were compared with the results of titrating with $TiCl_3$. The mean error was $\pm$ 0·8%. This method cannot be used for dyes that do not form insoluble salts quickly and quantitatively, e.g. Azo Fuchsine B (C.I. 16550). Attempts to replace benzidine by $CoCl_2$ or *p*-leucoaniline are described. C.O.C.

Paper Chromatography of Carotenes
J. Chromatography, **5** (June 1961) D34

R_f values of α-, β-, γ- and ζ-carotene (C.I. Natural Yellow 26) phytofluene, neurosporene, and lycopene (C.I. Natural Yellow 27), determined with paper impregnated with 20% activated alumina and using light petroleum (60–80°C) and light petroleum–benzene (80 : 20 v/v) mixture as solvents are given from data by A. Jensen, *Acta Chem. Scand.*, **14**, 2051 (1960). F.J.

Paper Chromatographic Separation of Coumarins and Furocoumarins
J. Grujić-Vasić *Mh. Chem.*, **92** (May 1961) 236–239

The R_f-values of 23 coumarins and furocoumarins are reported for six solvent mixtures, viz. isoamyl alcohol satd. with H_2O— n-butanol–ethanol–conc. aq. NH_4OH (4 : 4 : 1 : 1), and n-propanol–H_2O (90 : 10, 80 : 20, 70 : 30, and 20 : 80). H.H.H.

Partition Chromatography of Food Dyes on Paper. I— Preparation of Solvent Systems
V. Sadini *Russ. chim.*, **12** (3) (1960) 17–26
Chem. Abs., **55** (12 June 1961) 11690

Lists the R_f values of a number of food dyes in the following systems: water–butanol–acetic acid, water–butanol–propionic acid; water–ethyl acetate–formic acid, water–ethyl acetate–acetic acid, and water–ethyl acetate–propionic acid. C.O.C.

Test for Chromium in Water-soluble Extracts
J. Thuau *Rev. tech. Industr. Cuir*, **52** (1960) 23
J. Soc. Leather Trades Chem., **45** (June 1961) 267

Water-soluble chromium may arise from black Cr-containing dyes. The extract is treated with charcoal, filtered, and partly evaporated. After heating in a muffle with Na_2O_2, the residue is taken up in water and tested for Cr by addition of lead acetate soln. and acetic acid. If Cr is present a precipitate of lead chromate is formed. M.T.

Photometric Determination of Iron in Dyes
L. M. Golomb and V. V. Karpov
Khim. Volokna, (6) (1960) 55–56
Chem. Abs., **55** (26 June 1961) 12860

Dye (0·5–1·0 g) is placed in a 50-ml flask, pure HCl (*d* 1·19) (25 ml) added, and the solution kept on a steam bath at 80°C until the dye is dissolved and air eliminated. The solution is then cooled to room temperature, HCl (25 ml) added, and the solution diluted to volume with distilled water. After standing for 30 min, the solution is filtered through a double paper filter. A portion of the filtrate is placed in a 100-ml flask, 20% sulphosalicylic acid (10 ml) added, and NH_4OH introduced until the colour of the solution ceases to change. The volume is then made up with distilled water. The Fe content of the solution is then determined spectrophotometrically. C.O.C.

Polarographic Determination of Lead in Rutile (C.I. 77891) and Lead, Cadmium and Copper in Zinc White (C.I. 77497)
E. A. Navyazhskaya and L. N. Flegontova
Lakokrasochnye Materialy i ikh Primenenie, (5) (1960) 75–78
Chem. Abs., **55** (12 June 1961) 11876

To determine Zn in rutile, fuse 1 g pigment with 5–6 g Na in a Pt crucible. Leach the melt with 20% HCl or H_2SO_4, transfer the solution to a 250-ml flask and neutralise with conc. ammonia. Add 25-ml conc. ammonia, 10–12 g NH_4Cl, and dilute with water. Transfer 10-ml of the solution to an electrolyser and add 3 drops of 1% gelatin and 0·5 g Na_2SO_4. Determine the polarogram by using the "addition method". To determine Pb in Zinc White dissolve 2 g pigment in 20% HCl, transfer to a 250-ml flask, add 2 g KCl or KNO_3, and dilute with water. Transfer 10-ml aliquots into 2 electrolysers, to the first one add 3 drops of 1% gelatin solution, remove oxygen with hydrogen, and take the polarogram. To the second electrolyser add 0·1–0·5 ml. standard Pb solution (1 mg Pb/ml) and 3 drops of 1% gelatin solution and take the polarogram. To determine Cd in Zinc White, dissolve 2 g in 20 ml 20% HCl, place in a 100-ml flask, neutralise with 25% ammonia, add 20 ml ammonia and dilute with water. Transfer 10-ml aliquots to two electrolysers and take the polarograms as described above. To determine Cu in Zinc White, dissolve 1–2 g pigment in 15–20 ml 20% HCl, neutralise with conc. ammonia, add 20 ml ammonia and 5 g NH_4Cl and dilute to 100 ml. Transfer 10 ml of the solution into an electrolyser, add 3 drops of 1% gelatin and traces of Na_2SO_3 and take the polarogram by the "addition method". These methods are accurate and the determination takes 2–3 h. C.O.C.

Chromatographic Classification of Blue Ball-point-pen Ink
A. Usai and L. Corazzi *Russ. chim.*, **12** (1960) 40–41
Chem. Abs., **55** (26 June 1961) 12882

The inks can be classified by chromatography on Whatman No. 1 paper using alcohol–acetone (1 : 1) for 24 h at 25°C and 75% r.h. Acetic acid, HCl, NaOH, and ammonia are inferior as eluants. C.O.C.

Qualitative Determination of Oil Content of Organic Coatings by Gas Chromatography
W. L. Zielinski, W. V. Moseley, and R. C. Bricker
Off. Dig. Fed. Soc. Paint Technol., **33** (May 1961) 622–634

Identification of oils that have not been excessively affected by heat-treatment, oxidation, or reaction with monomers is straightforward. Heat-treatment, liming, or blowing of an oil leads to molecular growth and an increase in viscosity; this is reflected in diminished percentages of polyunsaturated ester peaks on chromatograms. In such cases, oil identification is assisted by a polymer estimation technique. These decreases are not usually shown in the case of oil-modified alkyds, epoxy cpd., and polyurethanes, but appreciable decreases are shown with oils copolymerised with cyclo- or dicyclo-pentadiene, or vinyl toluene, or reacted with maleic anhydride. An infrared study of the air-drying of tung oil esters shows

that there is an increase in carbonyl and peroxide formation concomitant with a decrease and shift in unsaturation absorption. J.W.D.

A new Rapid Embedding Method for the Preparation of Fibre Cross-sections
H. Braun and M. Monshausen
Melliand Textilber., **42** (June 1961) 662–666

A survey of the methods in common use for embedding hard and brittle textile materials is followed by the description of a new rapid procedure employing versamide, which is particularly suitable for the purpose. Versamide is a new type of polyamide obtained from the condensation of di- and tri-merised unsatd. fatty acids (preferably linoleic acid) with polyamines. There are 17 illustrations of cross-sections of various fibres and materials. H.H.H.

Measurement of Molecular Orientation in Fibres by Acoustic Methods
W. W. Moseley
J. Applied Polymer Sci., **3** (May–June 1960) 266–276

If the velocity of sound in completely orientated and completely disorientated fibres of a given material is known, then the orientation of a fibre of the same material can be calculated from the velocity of sound along it. P.T.S.

Sandocryl Dye Staining as an Optical Microscopic Technique for Identifying Acrylic Fibres
K. Kato *Text. Research J.*, **31** (June 1961) 578–580

A staining solution of Sandocryl Black NBLC–1 (0–2%), $CuSO_4 \cdot 5H_2O$ (0·2%), and copper wire mesh (5 g/l.), brought to pH 3 with phosphoric acid, readily stains acrylic fibres but leaves most other fibres unstained, with the exception of some protein fibres, e.g. wool, which are dyed to a dark colour. The microscope slides carrying the fibre sections are boiled in the stain for 30 min and then thoroughly rinsed with water. Viscose rayon or other fibres treated with acrylic polymers are readily detected by this method. Cellulose fibres which remain unstained do become more or less stainable if they are cyanoethylated. C.O.C.

Analysis of Fibre Blends— II
T. Szentpály, R. Szentpály, A. Stark, and E. Farkas
Faserforsch. und Textiltech., **12** (June 1961) 294–297

Methods previously described (*ibid.*, **11** (1960) 189; J.S.D.C., **76** (1960) 616) are used in the analysis of two- and three-component fibre mixtures. The accuracy of the methods is established and certain modifications are suggested. W.R.M.

Analysis of Mixtures of Polyester Fibres with Cotton or Viscose Rayon (Tentative Textile Standard No. 67)
J. Textile Inst., **52** (June 1961) P272–P275

The sample is extracted with light petroleum and then water or, if a special finish is present, this is removed by some other method. About 0·5 g of this pretreated material is dissected, cut into ca. 25-mm lengths, dried, and weighed. It is then treated with 100 ml of 75% (w/w) sulphuric acid for 1 h at 50 ± 5°C, the solution is filtered, and the residue is washed with water, dilute ammonia solution, and water again, and finally dried and weighed. This weight, with appropriate corrections, is the amount of polyester fibre in the sample. P.B.S.

Estimation of Degree of Set of Crimped Polyamide Yarns
K. Schwertassek and G. Vyskocilová
Textil Praxis, **16** (Apr 1961) 394–398

Modified continuous-filament polyamide yarns are prepared by false twist, conventional, stuffer box, and edge crimping. Differences in the degree of set can be ascertained by the critical soln. temp. and iodine absorption methods. Examples are given which illustrate how these parameters are affected by oil content of the yarns, type of spool used for setting (low heat-conducting ceramic are worse than metal tubes), temp. and duration of steaming. L.A.T.

Microscopic Examination of Mechanical Damage in Textiles
N. Bigler *S.V.F. Fachorgan*, **16** (Apr 1961) 236–240

Three examples of mechanical damage, accompanied by filamentation, and brought about by incorrect conditions of calendering and embossing, are described and illustrated by 9 photomicrographs. L.A.T.

Laboratory Methods for Measuring Washing Action
J. Radder and A. de Vlieger
Tex, **20** (June 1961) 297–305

Literature survey with 110 references. It is concluded that at present there is no method available which can be recommended as a standard. The most that a laboratory method can do is to differentiate between good and poor products. R.A.

Measurement of the Relative Coefficient of Visible and Infrared Reflection from Dyed Fabrics
D. Rydzewska *Prace Inst. Włók.*, **9** (1959) 17–38
Chem. Abs., **55** (12 June 1961) 11856

Measuring the relative coefficient of the visible and infrared reflection with a Zeiss monochromator makes possible evaluation of dyes and of their fastness to light and heat. C.O.C.

Measurements of the Spectra of Oxidation Dyeings
H. Wilmsmann
J. Soc. Cosmetic Chemists, **11** (1960) 560–571
Chem. Abs., **55** (12 June 1961) 11856

Data obtained by use of the Zeiss Elrepho spectrophotometer are given for oxidation dyeings on wool, buffalo hair, and human hair. The Kubelka–Munk function is valid for oxidation dyeings at low concentrations. C.O.C.

Experiments on the Removal of Water adhering to the Surface of Rainproofed Test Samples by Hydroextraction
H. Bundesmann and I. Matuschke
Melliand Textilber., **42** (May 1961) 561–565

Tests involving centrifugal extraction of fabric specimens after rainproofing trials have been carried out as a means of determining the amount of water removed from the surface of the material. The results have been compared with those obtained by the conventional methods of shaking by hand, and by the use of the English shaking apparatus. Particular emphasis is placed on the reproducibility of the results obtained by the various techniques. Three methods of hydroextraction were used in which (1) the sample hung freely on a vertically rotating axis (a simple laboratory stirrer), (2) the sample was mounted on a vertically rotating cylinder, and (3) the sample was mounted on a horizontally rotating disc. I.G.L.

Behaviour of Antistatic-finished Textiles towards Wearing and Washing Tests— II
E. Wagner *Chemiefasern*, **11** (June 1961) 410–412

The results of I (cf. *ibid.*, Mar 1961) on the use of antistatic agents and the influence of atmos. humidity on the discharge process are assembled, and the fastness of the finished textiles to washing and hot pressing recorded. H.H.H.

Factors Affecting the Determination of Carboxyl Groups in Cellulose
E. Sjöström and P. Haglund *Svensk Papperstidning*, **64** (15 June 1961) 438–446 (*in English*)

Bleached sulphite pulp, dissolving pulp, and bleached cotton linters were washed with dil. HCl. Effluent free from cations was obtained only after 2 weeks' contact time. No physical adsorption of magnesium chloride was observed. Mg is bound to the carboxyl groups by both valencies and neither mixed nor basic salts are formed. A method for determining carboxyl groups by Mg ion exchange is described and compared with existing methods. R.A.

Solvent Extraction of Leather
A.L.C.A. Vegetable Leather Subcommittee
J. Amer. Leather Chem. Assocn., **56** (May 1961) 213–221

Statistically planned determinations involving four different leathers, four solvents (petroleum ether, n-hexane, $CHCl_3$ and CCl_4), and four laboratories are analysed. The largest variance component obtained for solvent was 3·7008 (equivalent to a standard deviation of 1·92% extractive), whilst the largest for laboratory was 0·1441 (equivalent to a standard deviation of 0·38% extractive). It is recommended that n-hexane be accepted as the chosen solvent; the laboratory method could then be studied in an attempt to reduce the contribution of the laboratory to the variability of the results. A supplementary analysis of the results shows that the CCl_4 points show less scatter with respect to the overall solvent

averages than the points corresponding to the other solvents. The effect is particularly marked with one leather and it is possible that this leather contained substances that interfered with the analysis by all solvents except CCl_4, but this is merely speculation. Much further work is required to establish the real significance of these findings. J.W.D.

Water Absorption (Static) of Leather

J. Amer. Leather Chem. Assocn., **56** (June 1961) 289

A.S.T.M. method D 1815–60T for determination of the amount of water absorbed by leather at 23 + 2°C by immersion under static conditions. It may be used on all types of leather, but is particularly adapted to vegetable-tanned sole leather. The results do not reflect the water absorption under dynamic conditions of flexing. M.T.

Preliminary Tests for the Development of Suitable Solvent Systems for Paper Chromatography of Varnish Resins

K. Weigel

Deutsche Farben-Zeitschrift, **15** (June 1961) 239–241

Testing details are reported for 29 solvents (alcohols, esters, ketones, glycol ethers, hydrocarbons, methylene chloride, and trichloroethylene) of the paper chromatography of varnish resins H53 and M86 and the artificial resin AW2. There are 12 illustrations of the types of behaviour observed. H.H.H.

Ultraviolet Spectra of *N*-Heterocyclic Systems. II— Spectra of 5–8-Hydroxyisoquinolines and Related Compounds (IV p. 509)
Studies on the Dyeing of Acrylonitrile Fibre (VIII p. 522)
Migration of Liquids in Textile Assemblies. IV— Penetration of Fabrics by Liquids (X p. 526)

XV— MISCELLANEOUS

Colour-order System Predicting Constant Hue

F. W. Billmeyer, J. K. Beasley, and J. A. Sheldon

J. Opt. Soc. Amer., **51** (June 1961) 656–666

The limitations of some existing colour systems, e.g. the Munsell system, are indicated. Colour gamuts are illustrated for the Munsell system at value 6, the MacAdam limits of maximum chroma at this value, and that which can be reached in transparent acrylic resins. The last is much greater than the gamut for the Munsell samples which, like most systems based on physical samples, is limited to lower chromas than can be reached in transparent media. The system (ω-space) proposed by Moon and Spencer was known to correlate well with Munsell spacing in the low chroma region and has been modified to give what is described as an Adjusted-hue colour-order system (Ω-space). The equations for the Adjusted-hue system are given in a form suitable for machine computation, the value co-ordinate being identical with value in the Munsell renotation system. Conformation of the system to visual perception of colour was explored in a preliminary fashion using transparent acrylic resin samples. The colours of these samples were measured on a G.E.C. recording spectrophotometer with a digital readout system. Visual observations under conditions of average daylight were made by both skilled and unskilled personnel. The results are tabulated and discussed by comparison with the Munsell renotation system in terms of the visual observations. A better approximation to visually perceived hue is obtained with the adjusted-hue system. E.COATES

The British Chemical Industry— From K.I.D. to Outer Seven

G. Brearley

Chem. and Ind., No. 27 (8 July 1961) 1000–1008

An account is given of the relation of the industry to the economic structure and organisation, both in this country and in Europe in particular. Encouragement of research and development is essential to ensure that Britain's sales of know-how in the international market always exceed her purchases. Co-operation with the chemical industries within both the "Six" and the "Seven" to press for the establishment of one free-trading area in Western Europe is also urged. J.W.D.

Future of the European Textile Industry

R. Delerive *Chemiefasern*, **11** (June 1961) 364–368

This shortened version of a lecture given in Barcelona by the author to the National Association of Textile Technologists deals with the diversity of outlook for different nations on account of the formation of the EWG and EFTA groups, and their hindrance to a European textile plan. Six factual tables are included and discussed in relation to textile consumption, the fibres involved, the distribution of textile machinery, wage standards, imports and exports. The need for a common policy on an international basis is stressed. H.H.H.

Ultrasonics— Applications to Dyeing in U.S.A.

C. Weimann *Teintex*, **26** (15 July 1961) 509–511

In the U.S.A., applications are generally limited to the dyeing of hydrophobic fibres, and work has been mostly on a small scale. Brief descriptions of equipment are given, and examples of reduction in treatment times. J.C.F.

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Dihydroxyacetone as a Skin-colouring Agent (IV p. 510)
Fundamental Processes in Fluorescence Phenomena (IV p. 510)

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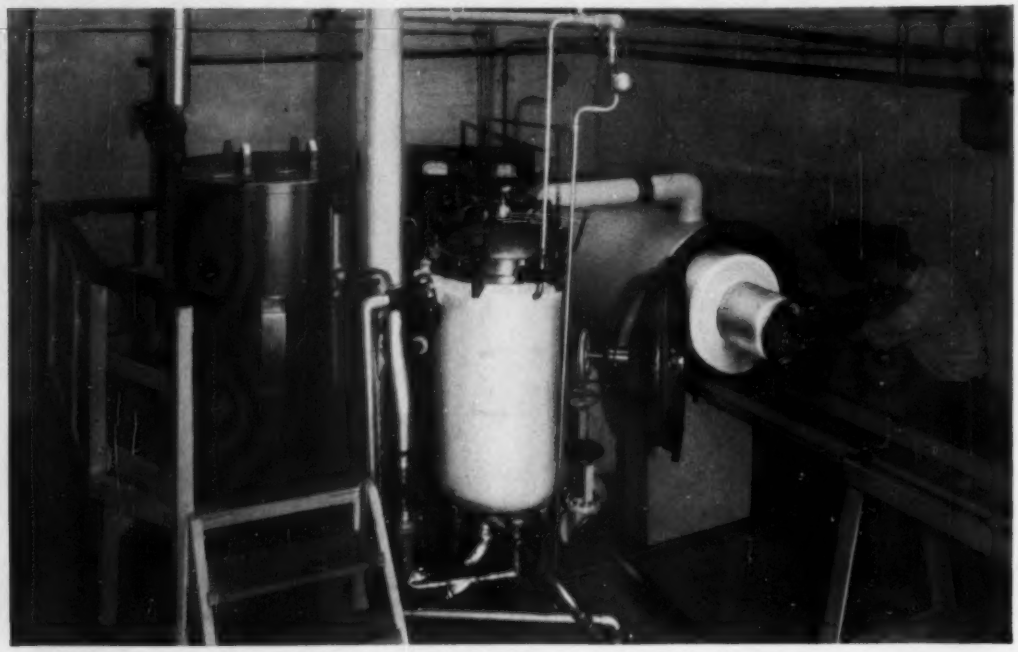

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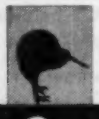

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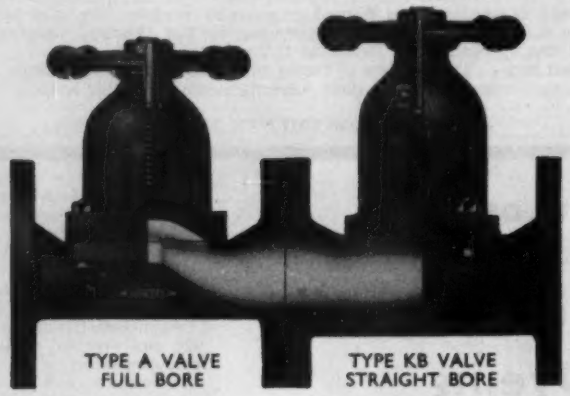

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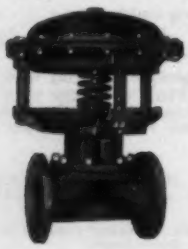

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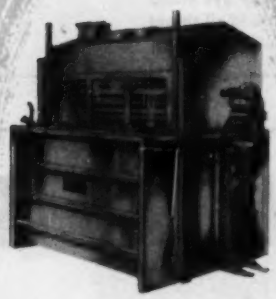

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Congress at Noordwijk, Holland 25–27 April 1962

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